

**CORRELATION OF LOCALLY-BASED  
PERFORMANCE OF ASPHALTS  
WITH THEIR  
PHYSICOCHEMICAL PARAMETERS**

**FINAL REPORT  
MARCH 1990**

**B. V. ENUSTUN  
S. S. KIM  
D. Y. LEE**

**IOWA DOT PROJECT HR-298  
ERI PROJECT 1942**

**Sponsored by the Highway Division of the  
Iowa Department of Transportation and the  
Highway Research Advisory Board**

**ENGINEERING RESEARCH INSTITUTE**

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"The opinions, findings, and conclusions expressed in this publication are those of the authors and not necessarily those of the Highway Division of the Iowa Department of Transportation."

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## ABSTRACT

Highway Research Project HR-298 was undertaken to study the relationships between the performance of locally available asphalts and their physicochemical properties under Iowa conditions with the ultimate objective of development of a locally and performance-based asphalt specification for durable pavements.

Physical and physicochemical tests were performed on three sets of asphalt samples including: (a) twelve samples from local asphalt suppliers and their TFOT residues, (b) six core samples of known service records, and (c) a total of 79 asphalts from 10 pavement projects including original, lab aged and recovered asphalts from field mixes, as well as from lab aged mixes. Tests included standard rheological tests, HP-GPC and TMA. Some specific viscoelastic tests (at +5°C) were run on (b) samples and on some (a) samples. DSC and X-ray diffraction studies were performed on (a) and (b) samples. Furthermore, NMR techniques were applied to some (a), (b) and (c) samples.

Efforts were made to identify physicochemical properties which are correlated to physical properties known to affect field performance. The significant physicochemical parameters were used as a basis for an improved performance-based trial specification for Iowa to ensure more durable pavements.

## I. INTRODUCTION

### 1.1 Background

Current specifications for asphalt cement contain limits on physical properties based on correlations established in the past with field performance of asphalt pavements. Recently, however, concerns have arisen that although current asphalts in use meet these specifications, they are not consistently providing the long service life once achieved.

There are a number of logically possible explanations of this situation:

(1) A considerable concern is associated with the recent world crude oil supply and the economic climate after the 1973 oil embargo which may have affected the properties of asphalt of certain origin (Hodgson, 1984). Blending several crudes, as routinely practiced in refineries to produce asphalts meeting current specifications, may have upset certain delicate balances of compatibility between various asphaltic constituents, which may manifest itself in their long-term field performance but not in original physical properties specified in the specifications (Goodrich et al., 1985; and Petersen, 1984).

(2) The increased volume and loads of traffic on highways, which have occurred over the decades, may have shortened the life span of pavements, indicating the necessity of revising specification limits and/or imposing new provisions to maintain desired durability.

(3) Inadequate mixture design, particularly poor gradation of aggregates, changing construction practices and improper use of additives may also be responsible for early deterioration of asphalt pavements (Anderson and Dukatz, 1985; and Hodgson, 1984).

(4) Specifications based only on physical properties of asphalts do not guarantee adequate performance.

While the performance of the asphalt pavements could be improved by judicious application of improved mix design techniques, more rational thickness design procedures, and better construction methods and quality control measures, the selection of asphalts based on performance-related properties, tests, and specifications is one of the key factors to durable asphalt pavements.

Highway Research Project HR-298 was approved by the Iowa Highway Research Board on December 2, 1986 to study the relationships between the performance of locally available asphalts and their physicochemical properties under Iowa conditions with the ultimate objective of development of locally and performance-based asphalt specifications for durable pavements. A Task I Report (Lee and Enustun, 1988) describing work performed and findings resulting during the first year of the study was submitted in January 1988. Progress Report No. 2 (Enüstün, Kim and Lee, 1989) describing work accomplished during the second year of the study was submitted in March 1989. This final report presents work performed during the third year and summarizes the findings and recommendations resulting from the three-year study.

### 1.2 Objectives

The objective of this study was to establish locally-based quality and performance criteria for asphalts, and ultimately to develop performance-related specifications based on simple physicochemical methods. In addition to physical tests, three of the most promising chemical methods (high performance liquid chromatography or HPLC, thermal analysis or TA and X-ray diffraction or XRD) were selected to analyze samples of:

- (a) Virgin asphalts and their residues from thin film oven tests and laboratory accelerated aging tests,



- (b) Asphalts extracted from selected field projects including plant mixes, core samples, laboratory mixes prepared using the virgin asphalts and project aggregates, both before and after they are artificially aged in the laboratory, and
- (c) Asphalts extracted from pavements with known performance records.

The results obtained would be analyzed to find the fundamental asphalt physicochemical parameters (such as viscosity, molecular size, micelle size, transition temperatures, temperature susceptibility, resistance to oxidative hardening, functional groups, etc.) which directly affect the field performance in Iowa.

On the basis of the laboratory-field-performance correlations, specifications would be formulated based on testing procedures which can be performed in the transportation materials laboratories of the Iowa DOT and ISU.

### 1.3 Program of Study

The ultimate objective of this study was to establish locally-based quality and performance criteria as a basis for asphalt specifications, in other words, the development of performance-based specifications for the state of Iowa.

This research was carried out in six tasks completed in three years. The specific tasks to be performed were presented in the research proposal and are shown in Figure 1.

## 2. EXPERIMENTAL

### 2.1 Materials

A total of 12 virgin asphalts commonly used in Iowa and obtained from two suppliers, as well as two sets of asphalt samples recovered from

pavements of known performance, were studied within the Task 1 (Lee and Enüstün, 1988). To accelerate the study and to gain an additional year of field performance record, it was decided to proceed with Tasks 2 and 3 at the same time. Ten hot mix field pavement projects were selected by the engineers of the Iowa Department of Transportation to represent a range of asphalt source, asphalt grade and type of projects in Iowa. The selected projects included four AC-5s, two AC-10s and four AC-20s. The projects consisted of two Interstate projects, three primary and five secondary highways, three of which were placed as surface, two as binder and five as base courses. A summary of these projects is given in Table 1. The project locations are shown in Figure 2.

From each project, one gallon of original asphalt cement, 30 to 50 lbs of virgin aggregates and 30 to 50 lbs of plant mix were collected. In addition, 2 to 3 core samples were taken after compaction. These samples were obtained between August and November 1988. Between September 1989 and January 1990, an additional 8 to 10 core samples were taken by Iowa DOT engineers at each project.

## 2.2 Methods and Procedures

From each of the 10 sets of field samples, the following asphalt cement samples were derived for physicochemical characterization:

- PA0: Virgin or original asphalt.
- PAR: Thin-film oven test residue following ASTM D1754.
- PO: Laboratory aged asphalt following pressure-oxidation procedure (20 atm of oxygen at 150°F for 46 hrs) developed under HR-124 (Lee, 1968 and 1974). This procedure was developed to simulate field in-service aging under Iowa climatic conditions.
- PO5: Laboratory aged asphalt following pressure-oxidation procedure (20 atm of oxygen at 150°F for 5 hrs)

- PM: Asphalt cement extracted and recovered from plant mix.
- PC: Asphalt cement extracted and recovered from core samples taken right after compaction.
- PC1: Asphalt cement extracted and recovered from core samples taken after one year of service.
- LM: Asphalt cement recovered from laboratory prepared hot mix following plant job mix formula using virgin aggregates and asphalt cement from the project.
- L35: Asphalt cement recovered from laboratory mix, compacted by 35-blow Marshall procedure and aged in oven at 140°F for 12 days (Goode and Lufsey, 1965 and Page et al., 1985). This procedure was developed to simulate in-service asphalt aging in pavements of high voids.
- L75: Asphalt cement recovered from laboratory mix, compacted by 75-blow Marshall procedure and aged in oven at 140°F for 12 days. This procedure was designed to simulate in-service asphalt aging in pavements of low void levels.

In the following discussion these asphalt sample codes will be preceded by a project number identified in Table 1.

2.2.1 Rheological properties: Penetration at 5°C (100 g, 5 sec), penetration at 25°C (100 g, 5 sec), penetration at 4°C (200 g, 60 sec), viscosity at 25, 60 and 135°C, and ring-and-ball softening point tests were performed on original (PA0), TFOT aged residue (PAR) and pressure-oxidation aged asphalts (PO and PO5), as well as asphalts recovered from plant mixes (PM), core samples (PC and PC1) and laboratory mixed (LM), compacted and aged samples at two void levels (L35 and L75). From these data penetration ratio (PR), penetration index (PI), pen-vis number (PVN), viscosity

temperature susceptibility (VTS), cracking temperature (CT), critical stiffness and critical stiffness temperature were calculated (The Asphalt Institute, 1981; Button et al., 1983). Based on viscosity data at 25°C, shear index (SI, the slope of log viscosity versus log shear rate plot) and complex flow (CF, the slope of log shear stress versus log shear rate plot) were also determined (Lee, 1974).

To correlate with low temperature field performance, the dependence of viscoelastic properties of selected asphalt samples, on their thermal history was studied at a low temperature. Newtonian viscosities and elastic shear moduli of these samples were determined at +5°C after cooling from +25°C, as well as after warming from a quenching temperature of -30°C. The instrument used for these measurements was a cone and plate viscometer modified to measure rotational displacements as small as 1/100 degree. Theoretical analysis of the experimental results showed that it was possible to estimate the Newtonian viscosity from the slope of the linear asymptotic section of the rotation versus time plot, and to estimate the elastic shear modulus from its intercept. The instrumentation, procedure and the theory were described earlier (Lee and Enüstün, 1988).

2.2.2 HP-GPC: High performance liquid chromatography (HP-GPC) is a technique by which the molecular size distribution of asphaltic components is determined by means of gels of selected pore sizes. As an asphalt solution is forced through a column of such gels, the Brownian motion compels the smaller molecules to take longer times than larger molecules to pass through the labyrinths of this column.

Recent reports from a Montana asphalt quality study using this technique have shown considerable promise and have led the Montana State Department of Highways to institute special provisions based on requirements based

on HP-GPC (Jennings et al., 1982, 1985 and 1988). While there were unresolved exceptions, it has been concluded that large molecular size asphaltic constituents contribute to low-temperature cracking of asphalt pavements. Other studies (Zenewitz and Tran, 1987; and Button et al., 1983) have related the amounts of small molecular size fractions to rutting and tender mixtures. Garrick and Wood (1988) reported correlations between asphalt chemical composition by HP-GPC and performance characteristics of asphalts and asphalt mixtures. Edler et al. (1985), in South Africa, found correlations between pavement deformation and bleeding and asphalts of certain molecular profiles as determined by HP-GPC. More recently, Goodrich (1988) found association between asphalts with wide distribution of molecular sizes as determined by HP-GPC, aging, and desirable mix characteristics with respect to low-temperature creep (rutting resistance). Work underway at Indiana, Kansas and Georgia Highway Departments (Bishara and Wilkins, 1989; Nouredin and Wood, 1989; and Caylor and Sharp, 1987) have also shown that the HP-GPC technique can be used as a reliable test to relate chemical composition and aging characteristics of asphalts. An interesting development in the field of GPC characterization of asphalts was reported by Pribanic et al. (1989) in which the wavelength of detection light is used as a variable. Using a multichannel UV-visible detector, this method makes it possible to obtain GPC chromatograms at eight different wavelengths simultaneously in one run. As wavelength scanning provides information on distribution of aromaticity and the functional groups over the molecular size range, this sophistication may prove to be valuable in asphalt characterization.

A high performance gel permeation chromatography (HP-GPC) system (Waters) was used during this study. The instrumentation and procedure were described earlier (Lee and Enüstün, 1988). To better characterize

the molecular size distribution of the asphalts, the HP-GPC profiles were analyzed by three different procedures: The normalized chromatograms were divided into three, four and eight slices following Montana State (Jennings et al., 1980), Iowa State (Lee and Enüstün, 1988) and Purdue (Garrick and Wood, 1988) procedures, respectively.

Twelve virgin asphalt (O) samples, their TFOT residues (R samples), six recovered core samples from seven-year old pavements, and 72 asphalt samples related to ten field projects were analyzed by HP-GPC.

2.2.3 Thermal analyses: Thermal analysis techniques have been used extensively by chemists to identify and characterize polymers. Breen and Stephens (1967) and Schmidt and Santucci (1966) recommended the use of glass transition temperature from thermal analysis data for predicting low-temperature cracking of asphalt pavements.

The glass transition point, which is known to depend to some extent on the scanning rate, is identified by a discontinuity in the expansion coefficient versus temperature plot, or in the specific heat versus temperature plot (Mascia, 1989). In actual practice, the former discontinuity is reflected to a thermomechanical (TMA) plot, i.e. a plot of linear dimension of the sample versus temperature, as a rounded break. The latter discontinuity manifests itself as an inflexion point in a differential scanning calorimetry (DSC) plot. Both methods are in use to determine glass transition points.

The application of DSC to asphalts has also revealed another transformation that takes place as they are heated from a low temperature. It is an endothermic transformation which may be interpreted as melting of crystallizable components (Noel and Corbet, 1970), or dissolution of these components in the liquid matrix (Albert et al., 1985), or dissociation

of agglomerates of asphaltene micelles (Lee and Enüstün, 1988). The presence of these species in an asphalt is believed to effect its low-temperature performance adversely. The enthalpy change associated with these transformations measurable by DSC (Albert et al., 1985) may measure the amount of these species in the sample.

In the present study twelve virgin asphalts and their TFOT residues and six recovered core samples were analyzed by a DuPont 1090 DSC instrument to determine their glass transition points, as well as endothermic enthalpy changes mentioned above.

Some of the samples in Task 1 as well as 79 samples related to 10 projects (Tasks 2 and 3) were also subjected to TMA tests to determine their glass transition points and other characteristics to be discussed below, using DuPont 943 TMA attached to the DuPont 1090 thermal analysis unit.

The procedural details of the DSC and the TMA methods were previously presented (Lee and Enüstün, 1988; Enüstün, Kim and Lee, 1989).

2.2.4 Nuclear magnetic resonance (NMR): It has been shown that valuable information can be obtained by  $^{13}\text{C}$  and proton NMR studies of asphalts regarding the average chemical functionality, e.g. carbon and hydrogen aromaticity, carbons with attached and non-attached hydrogen, as well as heteroatom (nitrogen and hydrogen functionality, in asphaltenes) for characterization of asphalts (Gerstein, 1983, 1986).

Four samples studied in Task 1 were subjected to  $^{13}\text{C}$  and proton NMR analyses, using a home-built solid state NMR spectrometer operating at 100 MH for  $^1\text{H}$  and 25 MH for  $^{13}\text{C}$ . This unit has extensively been used for studies of pyrolyzed pitches and coals supplied by Mobil Oil Research, the Argonne Coal Bank, and Iowa and German coals.

To fingerprint the heteroatom functionality by NMR, labeling with a ligand containing phosphorus was attempted.

Solution  $^{13}\text{C}$  NMR was employed for the two recovered Task 1 field asphalts, two Task 2 asphalts, and n-pentane insoluble asphaltenes of the Task 2 asphalts. For this purpose, Bruker WM-200 was used operating at 50 MHz for  $^{13}\text{C}$ . This unit is a research grade multi-nuclear NMR spectrometer for both routine and long-term experiments. Relaxation constant,  $T_{1\rho}$  (Gerstein, 1986) was measured by proton NMR using Bruker MSL-300, a high performance dedicated solid-state NMR spectrometer operating at 300 MHz for  $^1\text{H}$ .

2.2.5 Water-sensitivity of mixes: On the one-year old core samples, pavement performance against moisture damage was evaluated by measuring resilient modulus (RM) and indirect tensile strength (ITS) before and after an accelerated Lottman conditioning procedure (Lottman, 1982). A set of randomly selected three cores among the 8-10 cores from each project were subjected to RM measurement followed by ITS measurement. Another set of three random cores were subjected to RM and ITS measurements after the Lottman conditioning procedure which consisted of 30 min. vacuum saturation at 26 in. Hg, followed by 30 min. submerging under water at atmospheric pressure, 15 hr at  $-0.4^\circ\text{F}$ , 24 hr at  $140^\circ\text{F}$  water bath, and 3 hr at  $77^\circ\text{F}$  water. RM was measured with a Retsina Mark IV resilient modulus device at  $77^\circ\text{F}$ , 0.33 Hz frequency, and 0.1 sec load duration (Schmidt, 1972). ITS was measured at  $77^\circ\text{F}$  and a loading rate of 2 inches per minute (Kennedy, 1977).

2.2.6 Aging of asphalts: Age hardening characteristics of asphalt samples were studied in the laboratory by use of three different aging procedures; thin film oven test (TFOT), Iowa durability test (IDT), and mix aging. TFOT simulates age hardening due to conventional batch mixing (Goodrich and Dimple, 1986). The IDT or pressure-oxidation procedure consists of two aging stages: TFOT to simulate hardening during hot-plant mixing followed by pressure-oxidation under 20 atm of oxygen at  $150^\circ\text{F}$  for oxidative hardening



during field pavement service (Lee, 1968 and 1974). In this study, two different durations of pressure-oxidation, 46 and 5 hour, were used. It was found that 46 hours of IDT on asphalt was equivalent to 5-year aging of in-service pavement under Iowa climatic conditions. The previous IDT data indicated that 5 hours of IDT on asphalt was roughly equivalent to one year of field aging. Aging characteristics of asphalts were determined based on a theory that the changes in physical properties of asphalt are hyperbolic functions of time and approach a definite limit with time. An equation to express the hardening of asphalts in the field has been suggested as follows (Brown et al., 1957):

$$\Delta Y = \frac{T}{a + bT}$$

$\Delta Y$  = change in physical property with time T

a, b = constants

1/b = the ultimate change of property at infinite time

Constants a and b, and the ultimate change, 1/b, were determined from the measured physical property changes after 5 and 46 hour IDT. By use of the above equation, physical properties after 10, 20, and 30 year field service were estimated.

The hardening of asphalt in a mix is believed to be affected by air void content, asphalt film thickness, characteristics of aggregate, and the durability of the asphalt. To examine the age hardening of asphalt in a mix, Marshall specimens were prepared by use of the same materials and job mix formula used at each project. To simulate asphalt aging in pavement of high and low void levels, mixes were compacted by 35 blows per side and 75 blows per side, respectively, and oven-aged at 140°F for 12 days, equivalent to eight years of in-service asphalt aging in pavement (Goode and Lufsey, 1965 and Page et al., 1985).

### 3. RESULTS AND DISCUSSION

#### 3.1 Rheological Properties

The rheological properties of the Task 1 samples were reported and discussed previously (Lee and Enüstün, 1988). Penetration and softening point data of the 79 asphalts from the ten pavement projects are given in Table 2. The variability and changes in asphalts due to hot mixing and laboratory aging as indicated by penetration are shown in Fig. 3. Viscosity data including shear index and complex flow at 25°C are given in Table 3. Shear index or shear susceptibility (the rate of change of viscosity with rate of shear) and complex flow (the rate of change of shear stress with the rate of shear) have been found related to the aging characteristics of asphalts and useful indicators for pavement performance (Kandhal et al., 1973; Kandhal and Wenger, 1975; and Lee, 1974).

Asphalt cements of high temperature-susceptibility may contribute to rutting at high pavement temperatures and cracking at low pavement temperatures. Temperature susceptibility of an asphalt can be evaluated by penetration ratio, PR (penetration, 4°C, 200 g, 60 sec/penetration, 25°C, 100 g, 5 sec), the Penetration Index (PI), the Pen-Vis Number (PVN) based on viscosity at 60°C or viscosity at 135°C, the viscosity-temperature susceptibility (VTS), and the Asphalt Class Number (CN). Lower PR, large negative values of PI, lower PVN and greater VTS indicate greater temperature susceptibility. The temperature susceptibility indices in terms of PR, PI, PVN, VTS and CN of the asphalt cement samples studied are given in Table 4. The variability and changes in PVN at 60°C of asphalts studied are shown in Fig. 4.

The characteristics most relevant to asphalt performance and indirectly specified in the current ASTM D3381 and AASHTO M226 specifications are

temperature-susceptibility and resistance to aging. These important properties are plotted in Fig. 5 in terms of PVN (both 60°C and 135°C) and viscosity ratio at 60°C due to thin film oven treatment. The basic intent is that the most desirable asphalts should be limited to the upper, left corner. These asphalts have the best combination of properties of high resistance to aging and low temperature susceptibility. The ten asphalts supplied in Iowa during the 1988 construction season appeared to be rather uniform and well within the specifications.

Low-temperature asphalt stiffness has been correlated with pavement cracking associated with nonload conditions. The low-temperature behavior of asphalts can be evaluated either by estimating the temperature at which asphalt reaches a certain critical or limiting stiffness (the limiting stiffness temperature) or by comparing the stiffness of asphalts at low temperatures and long loading times (The Asphalt Institute, 1981; Kandhal, 1978).

Table 5 presents the results of estimated low-temperature cracking properties of the 79 asphalts from the ten projects. The properties include cracking temperature (CT), temperature corresponding to asphalt thermal cracking stress of 72.5 psi ( $5 \times 10^5$  Pa), based on penetrations at 5°C and 25°C, temperature of equivalent asphalt stiffness of 20,000 psi at 10,000 sec loading time (TES), estimated stiffness at -23°C and 10,000 sec loading time (S23), and stiffness at -29°C and 20,000 sec loading time (S29).

Goodrich (1988) found excellent correlation between low-temperature penetration (4°C, 200 g, 60 sec) and limiting stiffness temperature or temperature at which asphalt reaches a critical stiffness (e.g. 20,000 psi at 10,000 sec loading time), defined as temperature of equivalent stiffness (TES) in this study. He also found strong correlations between penetration at 4°C (200 g, 60 sec) and mix flexural fatigue life. Penetration at low temperature appears to be a good indicator for low-temperature cracking

and fatigue of asphalt and has been recommended as a criterion for cold climate asphalt specifications.

Statistical values of all properties of the ten asphalts were presented earlier (Enüstün, Kim and Lee, 1989), to show the variability of asphalts supplied in Iowa during the 1988 construction season. While, due to the small sample sizes, these data must be viewed with caution, and the significance of the variability must be interpreted and correlated with performance data, the following general comments can be made:

- There were larger variabilities in AC-20 asphalts than in AC-5 asphalts.
- Within each asphalt grade, there were more differences in stiffness at low temperatures, PI, class number, and PVN, than in cracking temperature, softening point and VTS.
- Laboratory aging seemed to increase the differences among asphalts of the same grade in some properties but decrease the differences in other properties.

The measured rheological properties of the Task 1 samples and the results of viscoelastic measurements at  $+5^{\circ}\text{C}$  with three virgin asphalts and two recovered core samples of known field performance as described in 2.2.1 were presented earlier (Lee and Enüstün, 1988). Here in Table 6 a summary of these measurements are given to show the striking differences between the responses of these samples to low temperature conditioning and the lapse of time.

All samples studied exhibit an increase in viscosity at  $+5^{\circ}\text{C}$  after cooling from  $+25^{\circ}\text{C}$  in various extents. This trend is more pronounced in more viscous asphalts. With virgin asphalts of high viscosity this increase is accompanied by a decrease in elastic modulus. Among the three samples conditioned at  $-30^{\circ}\text{C}$ , the effect of low temperature on viscoelastic

properties of SC-S is drastically different from those of the other two samples. In this respect, while the samples J05 and WR-S resemble each other, the viscosity and elastic modulus of SC-S drop drastically. The latter sample is known to have had a poor low-temperature field performance. This type of viscoelastic tests to be run on aged samples may, therefore, prove to be rewarding for predicting their low temperature performance. This method of testing turned out to be the only one to differentiate conclusively the Sugar Creek sample from the Wood River sample.

### 3.2 Chemical Properties

3.2.1 HP-GPC: The results of HP-GPC analyses of 12 virgin asphalts, their TFOT residues and six recovered core samples mentioned in 2.2.2 were presented in the Task 1 Report (Lee and Enüstün, 1988). The asphalt samples related to ten field samples were also analyzed by HP-GPC, and the normalized chromatograms of these samples were presented in Report No. 2 (Enüstün, Kim and Lee, 1989). For better chemical characterization of these samples, as well as those related to 12 original asphalts of Task 1 of the present project, the chromatograms were analyzed by dividing them into four slices as suggested earlier (Lee and Enüstün, 1988) and eight slices as practiced by Garrick and Wood (1988), as well as three Montana type slices (Enüstün, Kim and Lee, 1989). The analytic data pertaining to 10 field samples, including asphalts recovered from one-year old core samples, are presented in this report in Tables 7-11. Discussions based on these data, regarding their correlations with other properties and their potential bearing on prediction of field performance, will be presented in Sections 3.4, 3.5 and 4.2. The other significant findings can be summarized as follows:

1. The LMS contents of the virgin asphalts, as defined by the Montana research group varied between 20.6 and 35.9%, are higher than the maximum allowable (16-17%) for the Montana climate (Jennings and Pribanic, 1988).

2. A weak correlation was observed between LMS content and temperature susceptibility inferring that the higher the LMS the less is the temperature susceptibility. This correlation is parallel to what has been found by Glover et al. (1986). They and other workers (Zenewitz et al., 1987; Button et al., 1983) have also found that the lower the LMS and higher the SMS contents, the higher is the asphalt tenderness. In view of the results of the Montana researchers, it is likely that for a given climatic zone there is an optimum range of % LMS for the best pavement performance; too high LMS causes low temperature cracking, too low LMS causes high-temperature rutting and tenderness problems.
3. Percent LMS is unidirectionally sensitive to TFOT; virgin asphalts suffer an increase of 1.2-14.4% in their LMS contents. Therefore, the HP-GPC technique can be used to monitor and predict aging, as also concluded by other authors (Brule et al., 1987; Bishara et al., 1989; Nouredin et al., 1989; Caylor et al., 1987). The earliest-eluted fraction may serve as a better index for this purpose, as it is even more sensitive to aging. It is likely that the age-ability, i.e. the increase in LMS rating, rather than the initial LMS rating is a performance predictor.
4. Since the higher the concentration and the larger the molecules of solutes of a solution, the higher will be its viscosity; qualitatively speaking, the fact that aging increases the LMS content of an asphalt and its viscosity at the same time may appear to be a logical consequence. However, the quantitative aspect of these mutual shifts deserves attention: In Table 12 the differences in % LMS and the log of viscosity at 25°C of the virgin asphalt samples from 10 projects, induced by 46 hrs oxidative aging (PO), are tabulated. Given in the last column of this table are the ratios between these changes. It

is interesting to observe that as far as this ratio is concerned, eight samples out of 10 resemble each other regardless of their type and source. However two of these samples (Projects 2 and 7) significantly differ from the others, as the said ratio for these samples is about half of that of the majority, again irrespective of their type and source. The physical meaning of this observation is the fact that the structural and/or compositional nature of the smaller molecular size components of some samples may tolerate the growth and overpopulation of asphaltene molecules more than others, before they severely harden. In this respect, the samples from Projects 2 and 7 are expected to be different performers than the others.

3.2.2 T.A.: Considering the suggested bearing on low-temperature susceptibility of asphalts (Breen and Stephens, 1967; Schmidt and Santucci, 1966; Noel and Corbett, 1970; Albert et al., 1985; Brule et al., 1987) the samples related to Task 1 of this project were studied by the DSC technique in the heating mode and the results were presented earlier (Lee and Enüstün, 1988), in terms of estimated glass transition point, the enthalpy of endothermic transformations and their location in the temperature scale. Following Albert et al. (1985) the latter transformations can be interpreted as dissolution of some crystallized components formed at low temperatures in the asphalt matrix. Since the presence of such components in asphalt may affect its low-temperature performance adversely, it is expected that the smaller the enthalpy change ( $\Delta H$ ), the better will be its low-temperature performance. It is noteworthy that the samples supplied by Jebro Inc. appear to have significantly higher  $\Delta H$  values than those supplied by Koch Asphalt Co.

A total of 79 samples of virgin, aged and recovered asphalts from the lab and plant mixes and cores from 10 different paving projects in Iowa,

were subjected to thermomechanical analysis (TMA) in the heating mode to determine their glass transition temperatures ( $T_g$ ) by this method as described in Progress Report No. 2. The results were reported earlier, including a reasonable correspondence observed between the values obtained by DSC and TMA (Enüstün, Kim and Lee, 1989). Figure 6 illustrates how the glass transition points were determined. Table 13 summarizes the TMA results including one-year old core samples (PC1).

During the final phase of the present project, in addition to  $T_g$  values, three other parameters associated with TMA thermograms were also determined for the above mentioned 79 samples. They are the following (see Fig. 6):

1. The slope of the initial straight line (ML) which measures the low-temperature thermal coefficient of expansion of the sample at the glassy state. This slope has been proposed as an index to predict the performance quality of an asphalt (Lee, 1972).
2. The slope of the nearly straight adjacent section of the plot at higher temperatures (MH), which measures the coefficient of expansion after glass transition.
3. The softening temperature ( $T_{sp}$ ) at which the displacement of the TMA probe reaches a maximum.

From Table 13 it can be observed that:

1. The glass transition temperature,  $T_g$ , of the original asphalts ranged from  $-34^{\circ}\text{C}$  to  $-22.5^{\circ}\text{C}$ , increasing with viscosity from AC-5 to AC-20.
2. In general, aging at high temperature (PAO  $\rightarrow$  PAR  $\rightarrow$  PC1) reduced  $T_g$ ,  $T_{sp}$ , ML and MH while aging at low temperature (PC1  $\rightarrow$  PO5  $\rightarrow$  PO) increased the thermal responses. In other words, a different aging mechanism resulted in a different trend of thermal responses. Projects 10 and 12 asphalts are presented in Figs. 7 to 10.



3. Among the asphalts recovered from one-year old cores, asphalt in the project 2 showed exceptional thermal responses as shown in Figs. 11 to 14. Theoretically it is expected that this asphalt would behave differently than the others.

Correlations between TMA parameters and other properties will be discussed in Section 3.5.

3.2.3 Nuclear Magnetic Resonance (NMR): The samples studied by NMR spectroscopy were the virgin asphalt sample J10-02-0, its oven treated version J10-02R and the core samples SC-Bi and WR-Bi from the Sugar Creek and Wood River projects, respectively. As reported earlier (Enüstün, Kim and Lee, 1989), their spectra indicate that:

- (1) Oven treatment decreases the amount of aliphatic quaternary carbon in the virgin sample.
- (2) The quaternary carbon content of the Sugar Creek sample is strikingly less than those of all other three samples.
- (3) The line-width of proton NMR spectra of the Sugar Creek sample is also significantly larger than those of other samples, meaning that this sample is more rigid than the others. It is significant to note that this asphalt was earlier identified as a poor performer.

From the efforts to ligate the asphalt samples with the phosphorus marker, it was concluded that the small amount of heteroatoms in asphalt made this technique not to be viable. Solution  $^{13}\text{C}$  NMR spectra of SC-Bi, WR-Bi, 7PAO and 7PO did not indicate any significant differences among them. The spectra of the n-pentane insoluble asphaltenes of 7PAO and 7PO showed no significant difference between them. Removal of the n-pentane soluble portion of asphalts only increased the intensities of aromatic peaks relative to aliphatic peaks. Effect of solvents was examined by using three different solvents (deuterated chloroform, deuterated tetrahydrofuran (THF), and

duetrated toluene), and no noticeable effect on  $^{13}\text{C}$  solution NMR was found. Measurement of relaxation constant,  $T_{1\rho}$  indicated that 7PO with 1.50 ms of  $T_{1\rho}$  was slightly harder than 7PAO with 1.40 ms of  $T_{1\rho}$ . However, this difference could not be considered as significant when the instrumental error was considered.

Due to the nature of the asphalt, a complex mixture of hundreds of thousands of different molecular structures, to find differences in chemical shifts or intensities among asphalt samples' NMR spectra for the purpose of asphalt cement characterization seems to be problematic.

### 3.3 Water Sensitivity of Mixes

Resistance to moisture-induced damage of one year old cores were evaluated by using retained tensile strength ratio and retained resilient modulus ratio after Lottman-accelerated moisture conditioning. As shown in Table 14, the cores taken from the project 11 showed the least resistance to moisture-induced damage in terms of resilient modulus and the indirect tensile strength retained. Also, the cores taken from the project 10 with the highest percent air void among those of all projects could be considered as susceptible to moisture damage in terms of indirect tensile strength retained.

### 3.4 Aging

Levels of aging or property changes due to thin film oven test (TFOT) were compared with those due to actual construction operations by comparing rheological properties of TFOT residues (PAR) and asphalt samples recovered from plant mixes (PM), cores taken right after construction (PC), and lab mixes (LM). In general, TFOT caused more hardening for soft asphalt (AC-5) than the harder asphalt (AC-10 or AC-20). In AC-20 asphalts, TFOT caused about the same hardening as hot mixing in terms of P5, P25, P4, and Vis 25. However, in other properties, TFOT caused more changes than the hot

mixing process. Examination of CF and SI revealed that TFOT residues showed less hardening (higher CF and lower SI) than asphalts mixed and recovered (PM, PC, LM).

Based on pressure-oxidation treatment for 5 hrs and 46 hrs, considered to be equivalent to 1 yr and 5 yr of field aging under Iowa climatic conditions, respectively, rheological properties and HP-GPC parameters were predicted for 10, 20, and 30 year field service, using the method suggested by Brown et al. (1957) as given in Tables 15 and 16 together with the ultimate properties. Predictive equations of penetration at 4°C and R&B softening point for AC-5 asphalts were plotted in Figures 15 and 16. In some cases, the prediction based on two point measurements resulted in mathematically impossible expressions. As can be seen from Table 15, the project 2, 3 and 7 asphalts would become stiffer than others beyond five years. In this study, it was found that the 2nd and the 7th size fraction (X2 and X7) of the HP-GPC profiles based on the 8-slice method were very closely related to most of the asphalt properties (see Section 3.5). Table 16 gives the predicted percentage of these two slices along with X1. The project 2 asphalt yielded high X1 and X2 and the lowest X7 value for long term prediction. This prediction indicates that this particular asphalt would become too stiff to perform well. The project 3 asphalt was predicted to give the highest X1 and the second lowest X7 after 10 year field service. The asphalt used in the project 12 resulted in the highest X2 at the ultimate stage. These three project asphalts could be categorized as susceptible asphalts, especially as far as low-temperature cracking properties are concerned.

Generally, the asphalt in a mix with high air void ages more than the asphalt in a mix with low air void. In this study, air voids of lab mixes compacted by 75 blows per side (L75 mixes) ranged from 3.37 to 7.11% and

air voids of the mixes compacted by 35 blows per side (L35 mixes) ranged from 6.57 to 9.69%. The L75 mixes had 0.58 to 4.08% less air voids than the L35 mixes. The overall relationship between air voids and asphalt age-hardening was not consistent. Only with AC-10 asphalts, it was observed that the higher the air void, the more aged was the asphalt.

The rheological properties of TFOT residues (PAR) and the recovered asphalts after one year field aging (PC1) are compared in Tables 2 and 3. The recovered asphalts of the project 4, 5 and 10 were found aged more than TFOT residues in all properties, while the recovered asphalts of the rest of the projects were aged less in terms of all properties except complex flow (CF) and shear index (SI). Goode and Lufsey (1965) pointed out that air permeability, air voids and asphalt film thickness were major factors affecting age hardening of asphalt pavement. Indeed, as seen in Table 14, the cores from these three projects showed the highest air voids among the projects of the same grade of asphalt.

In terms of rheological properties, asphalts pressure-oxidized for 5 hours (PO5) aged more severely than PC1 asphalts. The order of aging within a project, in general, was  $PA0 < PC1 < PO5 < PO$  in terms of rheological and HP-GPC properties. However, in thermomechanical analyses, only three aged asphalts showed a general trend as  $PC1 < PO5 < PO$  in terms of glass transition temperature ( $T_g$ ), softening temperature ( $T_{sp}$ ) and the lower temperature expansion slope (ML), as shown in Figures 17, 18 and 19.

Previous studies have shown that aging characteristics of asphalt during high temperature plant mixing (short-term), as with thin film oven tests, may or may not reflect the aging characteristics of the asphalt during low temperature aging in pavement (long-term), as with low temperature pressure-oxidation tests. This is demonstrated in Figure 20, in which the long term rate of age-hardening or long term aging index, defined as the

ratio of viscosity of 46 hr pressure-oxidized asphalt (PO) at 60°C to viscosity of TFOT residue (PAR) at 60°C, were compared with short term aging index, which is the ratio of viscosity of PAR at 60°C viscosity of original asphalt (PAO) at 60°C. The project 2 asphalt showed a drastic difference between short-term and long-term aging indices; it was the least aged in terms of short-term aging index and the most aged in terms of long-term aging index.

### 3.5. Correlations

In this section the discussion will be confined to correlations between physical properties, TMA and HP-GPC parameters of all samples related to the 10 field projects.

Regression analyses were performed among physical parameters, TMA parameters, and molecular weight profile derived from HP-GPC tests. Table 17 gives results of regression analyses between TMA parameters and HP-GPC parameters for 73 samples. It indicates that TMA parameters, Tsp and ML correlate with HP-GPC parameters by the 8-slice method fairly well. Results of multiple linear regression between physical properties and TMA parameters are given in Table 18. These results show significant correlations between TMA parameters and almost all the physical properties with the exception of temperature susceptibility. Among the temperature susceptibility parameters only penetration ratio (PR) and pen-vis number at 60°C (PVN60) significantly correlate with Tsp and ML. Tsp correlates well with both rheological and low-temperature properties, while Tg correlates well with low-temperature properties. The initial slope, ML, appears to be a strong predictor of rheological properties. Results of multiple linear regression analyses performed between physical properties and HP-GPC parameters are given in Table 19. Molecular size distribution is best characterized by the 8-slice method thus correlating well with almost all physical properties.

Our results support findings by Garrick and Wood (1988) and Price and Burati (1989) that molecular size profiles derived from HP-GPC data can be used to predict many of the physical properties. HP-GPC parameters do not however correlate well with temperature susceptibility in terms of PI, CN, and VTS.

Since TMA and HP-GPC parameters were significantly correlated with rheological properties and TMA and HP-GPC parameters had less significant correlation with each other, it was worthwhile to try to treat these two sets of parameters as independent but complementary variables to correlate with rheological properties. Table 20 gives a summary of regression analyses performed as such on physical properties against TMA and HP-GPC parameters combined. These regression analyses give considerably higher r square values than the regression analyses using TMA or HP-GPC parameters alone. Figures in Appendix II compare the actual physical properties with predicted properties from regression equations which are given in Appendix I. Among TMA and HP-GPC parameters, more significant parameters were identified by using the step-wise linear regression technique. While, as indicated above, Tsp among TMA parameters was significantly correlated with rheological properties, X2 and X7 among HP-GPC parameters most predominantly control the rheological and low-temperature properties.

#### 4. PROPOSED TRIAL ASPHALT SPECIFICATIONS FOR IOWA

##### 4.1 Rationale

The selection of the proper grade of asphalt for a given paving project must be based on consideration of climate (temperature), traffic, thickness of the layer and the prevailing construction conditions. The selection of an asphalt within a given grade must be based on temperature susceptibility and durability. The temperature susceptibility of asphalt influences the mixing, placing and compaction of the paving mixture as well

as the high and low temperature performance of the pavement. Durability of asphalt or asphalt's resistance to hardening and aging, during construction and in-service, affect the pavement life.

In spite of the fact that a great deal of research has been accomplished with regard to asphalt properties and pavement performance, there is still no agreement as to the critical characteristics of asphalt that control the performance of asphalt pavement except hardness. Hardening of asphalt may cause the mixture to become brittle and susceptible to disintegration, cracking and moisture damages. The mechanisms contributing to asphalt hardening are many and varied. It is generally accepted that the major causes of asphalt hardening are volatilization during mixing at high temperatures (short term) and oxidation during service in pavement (long term). Current asphalt specifications, while containing requirements for indirect control of temperature susceptibility and asphalt hardening during hot-plant mixing, have no control over long-term durability.

As early as 1892, paving engineers became aware of the fact that the durability of the asphalt pavements often depended on the ability of asphalt cement in resisting changes and that it must be capable of cementing together the aggregate and it must be elastic and in no way brittle. They realized, shortly after the first asphalt pavements were constructed in the United States, that test methods were needed to measure the properties of asphalt and realistic specifications were necessary to assure the use of durable asphalts (Welborn 1984).

Many durability test and evaluation methods have been used since the early 1900's (Tables 21 and 22). Of the various methods proposed for evaluating the durability of asphalts, only the thin-film oven and rolling thin-film oven test methods have been adopted as standard by ASTM and AASHTO. Nearly all the agencies have adopted one of the methods as a means of measuring potential hardening of asphalt during hot-plant mixing.

Methods are available in the current specifications to predict the properties of asphalt at the time of construction; however, they do not provide adequate information on changes in properties during service in the pavement. This is particularly true of the low-temperature properties and long-term aging characteristics. In order to develop performance-related specifications for asphalt pavements, there is a real need for the development of a rational method to predict long-term asphalt durability during service in pavements. A trial specification based on Iowa pressure oxidation test is proposed. It is of significance to note that (a) researchers associated with SHRP projects consider pressure oxidation procedure the most promising for long-term aging evaluation, (b) the SHRP A002A team at Laramie is evaluating our test procedure for adoption and, (c) a performance-based asphalt specification similar in concept is being contemplated by SHRP as a major product of its asphalt research.

The specification is proposed with the belief that: (a) there is a wide range of differences in durability among paving asphalts; (b) current specifications do not discriminate nondurable asphalts; (c) simple methods can be developed to evaluate the relative durability of asphalts, and (d) pavement service can be improved by more durable asphalts.

The key element of the proposed specification is the Iowa Durability Test (IDT) developed under HR-124 based on the following rationale:

1. Hardness of asphalt in the pavement is the one property most closely associated with pavement performance. Therefore the extent and rate of asphalt hardening is considered to be indicative of the relative durability of asphalt.
2. The hardening and other pertinent changes that may occur in asphalt in an asphaltic concrete mix take place in two stages under two entirely



different environments or conditions: hardening during short periods in the mixer at higher temperatures and higher rates, and hardening during longer periods of road service in pavement at relatively lower temperatures and lower rates. The hardening mechanisms and effects in these two stages are believed to be quite different.

3. Any realistic durability test for asphalt should include consideration of the two stages of hardening processes of asphalt in their logical order and of their differences in mechanisms and effects.
4. Hardening during the mixing process may be simulated and predicted in the laboratory by the thin film oven test (ASTM D1754; AASHTO T179). Additional hardening and other changes in the asphalt in service may be simulated by laboratory pressure-oxidation tests at road service temperature on residue of the TFOT (Iowa Durability Test).
5. A definite correlation may be established, at least on a local basis, between field hardening and performance of asphalt and laboratory-accelerated hardening during a logically conceived and realistic durability test. The asphalt hardening in the field in terms of years could be reasonably predicted in terms of hours or days.
6. Results from this and more recent other studies confirm that chemical or compositional factors have a major impact on the performance of asphalt. While specifications based solely on chemical composition would be costly and difficult to implement, a rational specification based on both short-term and long-term accelerated aging test, containing time-honored physical tests and temperature susceptibility control, coupled with minimum chemical and low-temperature requirements is both desirable and feasible.

It must be recognized that selection and/or establishment of durability criteria and critical values of critical properties are complex problems

and require long-term field performance data. Therefore the recommended tests, properties and limits must be considered preliminary and tentative. When additional performance data become available these tests and limits should be modified and refined to reflect Iowa conditions. However, it is believed that the Iowa Durability Test (IDT) procedure can be considered logical, realistic, and simple; the equipment is inexpensive and the procedure is reproducible and effective. If parameters, tests and critical values of asphalts are properly selected, the results of this investigation can be applied to asphalt specifications for assurance of durable paving asphalts.

#### 4.2 Proposed Trial Specification

Many researchers have proposed physical properties of asphalts and their critical limits for acceptable pavement performance. Some of these properties are penetration at 4°C and 25°C, R&B softening point, viscosity at 25°C, shear index, pen-vis number and stiffness at a low temperature, as summarized in Table 23. No critical values for penetration at 4°C (P4) exists in the literature. Studies showed significant correlation between P4 and low temperature properties (Goodrich 1986 and 1988, Kemp and Predoehl 1981). Stiffness at -23°C and 10,000 sec loading time (S23) was correlated with P4 to estimate its critical value. Indeed, as presented in Figure 21, P4 was significantly correlated with S23 with  $r^2 = 0.906$  in the present study. The critical value of P4 corresponding to stiffness of 20 ksi was found as 5. Although, due to insufficient field performance data, direct correlations between HP-GPC and TMA parameters and field performance are not yet available at this stage, critical values of HP-GPC and TMA parameters were indirectly estimated from correlations with the performance related physical properties as given in Table 23. Since changes of asphalt properties after 5 years of aging are usually small, the critical values

discussed above are recommended as limiting values in specification for an asphalt pressure-oxidized for 46 hours at 150°C and 20 atm oxygen.

To prevent a low-temperature asphalt pavement transverse cracking problem, use of cracking temperature criteria in pavement design has been suggested (The Asphalt Institute, 1981). In this design method, the cracking temperature determined from penetration at 25 and 5°C should be lower than the pavement design temperature. For Iowa climate, a minimum pavement temperature of -35°C was estimated. The minimum penetrations at 5°C for AC-5, AC-10 and AC-20 and this cracking temperature were determined from the cracking temperature nomograph as 10, 8 and 7, respectively.

To assure long term durability, it seems necessary to limit the long-term aging index, i.e. ratio of viscosity at 60°C after pressure-oxidation for 46 hours to viscosity at 60°C after TFOT, as defined in Section 3.4. The long term aging indices for the 10 project asphalts range from 2.9 to 3.5 except the project 2 asphalt of which the long term aging index was as high as 6.3. Based on this observation, a critical long term aging index of 5 is tentatively suggested. The proposed specification, based on the pressure oxidation test and existing AASHTO M226, Table 2, is given in Table 24. Some of the limiting values can be increased or decreased as more field performance data become available.

Usefulness of the LMS fraction in the original asphalt and its amount of change after laboratory aging to predict asphalt pavement performance has been recognized (Jennings et al., 1982, 1985 and 1988). Although they are not included in the trial specification due to insufficient performance data, it is recommended that they be included when long-term performance data become available.

## 5. SUMMARY AND CONCLUSIONS

Three groups of asphalt samples were tested during this investigation including: (a) twelve samples from local asphalt suppliers and their TFOT residues, (b) six core samples of known service records, and (c) a total of 79 asphalts from 10 pavement projects including original, lab aged and recovered asphalts from field mixes and pavement cores, as well as from lab aged mixes. They were studied by physical and physicochemical tests including standard rheological tests, HPGPC and TMA. Some specific viscoelastic tests (at +5°C) were run on (b) samples and on some (a) samples. DSC and X-ray diffraction studies were also performed on (a) and (b) samples. Furthermore, NMR techniques were applied to some (a), (b) and (c) samples.

Efforts were made to identify physicochemical properties which are correlated to physical properties known to affect field performance. The significant properties formed the basis for a recommended performance-based trial specification for Iowa to ensure better pavement performance.

The conclusions of a general nature are summarized below:

- (1) Within each viscosity grade of asphalts available in Iowa and meeting AASHTO Specification M226, there were differences in temperature susceptibility between the samples supplied by different suppliers and between samples from the same supplier over time.
- (2) Distinctly different GPC chromatograms, TA results and X-ray patterns were obtained among asphalts of the same grade, same supplier, but supplied at different times.
- (3) The strikingly different effect of a cold shock (-30°C) on the viscoelastic properties of the core sample from the surface course of the Sugar Creek project from the other samples might have an important bearing on its poor field performance.

- (4) Elastic shear modulus measured at a low temperature may be correlated to low temperature field performance.
- (5) No decisive correlation is observed between GPC, DSC and X-ray results.
- (6) In contrast to thermal analytic behavior and x-ray diffraction spectra, LMS rating is found to be conclusively and unidirectionally sensitive to aging and when analyzed over the entire spectra of molecular size distribution by the 8-slice method, can be used to predict behavior and performance of asphalts. However, for specification purposes, both original and lab-aged asphalts must be tested.
- (7) The endothermic peaks on DSC thermograms indicate the presence of crystallizable components, while the LMS rating measures the presence or tendency of gel formation. Therefore, the extent of these peaks ( $\Delta H$ ) may be used to evaluate the low-temperature-susceptibility of asphalts, together with their LMS rating.  $\Delta H$  values are on the whole more pronounced in virgin asphalts of Jebro origin than Koch asphalts.
- (8) Asphalts used in the 1988 construction season from a limited number of sources in Iowa showed differences not obvious by either physical or physicochemical tests alone. For example, the asphalt used in Project 7 had a large percent increase in LMS due to aging, but not reflected by changes in physical properties, e.g. viscosity ratio. On the other hand, A.C. No. 11 had a high viscosity ratio after TFOT aging, but this was not reflected in an increase in LMS.
- (9) Aging, both in the field and in the lab, is accompanied by hardening, reduction in temperature susceptibility by most measures, an increase in shear susceptibility, decrease in complex flow, increase in temperature for limiting stiffness, increase in stiffness at low temperatures, increase in LMS and a decrease in SMS. For some asphalts, aging characteristics during high-temperature (short-term) and service temperature (long-term) were very different.

- (10) The glass transition points determined by TMA are in general agreement with those determined by DSC, and correlate fairly well with low-temperature cracking properties.
- (11) Both TMA and HP-GPC parameters correlated well with physical properties. Tsp correlates well with both rheological and low-temperature properties, Tg correlates well with low-temperature properties and ML is a strong predictor of rheological properties. Molecular size distribution based on HP-GPC and the 8-slice method can be used to predict many of the physical properties.
- (12) While TMA parameters and HP-GPC parameters did not correlate well, physical and low-temperature properties can be predicted by combinations of these two sets of parameters, especially using Tsp, ML, X2 and X7.
- (13) The relative significance of the more than 30 physicochemical parameters in predicting the field performance can only be established through correlation with field performance data. It is possible that the predictive equation must contain both physical and physicochemical parameters.

## 6. RECOMMENDATIONS

1. A tentative specification for paving asphalts, including durability requirements based on IDT, as well as chemical and low-temperature requirements, is recommended.

2. To improve the tentative specifications (weaknesses due to lack of sufficient data on critical values of critical properties under Iowa weathering and traffic conditions), continued observations and tests of the 10 pavements are recommended. continued study of the 10 pavements is needed to refine the critical specification values for Iowa conditions for a truly performance-based asphalt specification.

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Table 1. Summary of field samples.

Project	County	AC source	Pavement
1	Monona	AC-10 KOCH, Algona 3/4" AGG. 70% gravel 30% crushed gravel	surface, S <sup>a</sup>
2	Story	AC-20 KOCH, Tama 3/4" AGG. 65% 3/4" crushed limestone 10% 3/8" chips 25% sand	binder, P <sup>b</sup>
3	Dallas	AC-20 KOCH, Dubuque 3/4" AGG. 50% 3/4" crushed gravel 35% 3/4" quartzite 15% concrete sand	surface, I <sup>c</sup>
4	Grundy	AC-5 KOCH, Dubuque 1/2" AGG. 70% 3/4" gravel 12% 3/4" crushed gravel 18% 1/2" crushed limestone	base, S
5	Hardin	AC-5 KOCH, Dubuque 3/4" AGG. 70% 3/4" gravel 30% 3/4" crushed limestone	base, S
7	Webster	AC-5 KOCH, Algona 3/4" AGG. 60% 3/4" crushed limestone 40% 3/4" gravel	base, S
8	Plymouth	AC-5 KOCH, Algona 3/4" AGG. 17% 3/4" wash rock 83% 3/4" pit run	base, S
10	Harrison	AC-20 JEBRO, Sioux City 3/4" AGG. 35% 3/4" quartzite 14% concrete sand 51% 3/4" crushed rock	surface, P
11	Harrison	AC-10 KOCH, Algona 3/4" AGG. 30% 3/4" limestone 30% 3/8" limestone 40% crushed gravel	binder, P
12	Pottawattamie	AC-20 KOCH, Omaha, NE 3/4" AGG. 50% 3/4" stone 35% 3/8" stone 15% sand	binder, I

<sup>a</sup>Secondary road.<sup>b</sup>Primary road.<sup>c</sup>Interstate Highway.

Table 2. Rheological properties - I.

sample ID	P5	P25	P4	S.P. C
(AC-5s)				
4PAO	19	181	64	41.5
4PAR	14	100	52	45.5
4PC1	11	98	35	44.6
4PO5	11	86	31	48.8
4PO	10	52	25	54.0
4PM	18	144	56	43.5
4PC	15	105	45	46.5
4L35	15	105	54	54.0
4L75	12	55	29	55.0
5PAO	18	191	68	41.5
5PAR	13	103	40	48.0
5PC1	11	83	33	49.3
5PO	11	53	25	54.0
5PM	19	156	61	45.5
5L35	12	77	39	51.0
5L75	13	86	37	50.0
7PAO	16	193	60	39.0
7PAR	12	94	38	43.5
7PC1	14	105	41	45.3
7PO5	11	84	32	49.3
7PO	10	46	24	56.0
7L35	17	105	44	45.5
7L75	14	91	39	50.0
8PAO	17	196	58	38.5
8PAR	13	95	39	46.5
8PC1	15	107	43	44.5
8PO5	11	83	30	50.2
8PO	10	46	25	56.0
8L35	15	88	36	49.5
8L75	14	84	42	50.0
(AC-10s)				
1PAO	8	82	29	47.5
1PAR	7	50	21	52.0
1PC1	7	52	21	51.4
1PO5	6	44	16	54.1
1PO	6	27	14	61.5
1PM	10	55	29	56.0
1L35	8	27	15	66.5
1L75	9	32	17	62.5

Table 2. (continued)

sample ID	P5	P25	P4	S.P. C
11PAO	15	133	44	44.0
11PAR	10	69	29	51.5
11PC1	11	91	36	49.0
11PO5	8	58	24	53.2
11PO	7	35	21	59.5
11L35	10	60	27	55.0
11L75	11	65	28	54.0
(AC-20s)				
2PAO	7	54	15	49.0
2PAR	6	38	17	55.5
2PC1	7	45	19	55.8
2PO5	5	36	14	52.6
2PO	6	25	14	67.0
2PM	7	35	18	59.0
2PC	6	30	17	61.0
2LM	7	39	19	60.5
2L35	5	31	16	60.5
2L75	7	35	16	58.5
3PAO	9	75	30	47.0
3PAR	8	48	22	54.5
3PC1	11	89	36	47.2
3PO5	5	41	16	55.8
3PO	6	26	14	63.0
3PM	9	41	22	58.0
3PC	9	40	24	58.0
3L35	7	30	16	66.5
3L75	6	33	18	61.5
10PAO	9	82	29	49.0
10PAR	7	47	19	50.5
10PC1	6	36	18	59.0
10PO5	5	40	16	56.5
10PO	5	24	14	62.5
10L35	8	32	19	65.0
10L75	10	81	30	48.5
12PAO	8	82	28	47.0
12PAR	6	47	20	53.5
12PC1	9	67	27	49.6
12PO5	5	40	15	56.2
12PO	4	23	14	63.0
12L35	9	54	21	56.0
12L50	10	65	27	53.0
12L75	9	51	24	54.0

P5: penetration @ 5C, 100g, 5sec; P25: penetration @ 25C, 100g 5sec; P4: penetration @ 4C, 200g, 60sec; S.P.: Ring & Ball softening point

Table 3. Rheological properties - II.

sample ID	VIS 25 poise	C.Flow	S.Index	VIS 60 poise	VIS 135 cSt
(AC-5)					
4PAO	1.50E+05	0.98	0.030	583	250.3
4PAR	7.20E+05	0.96	0.025	1574	368.6
4PC1	9.00E+05	0.96	0.065	1730	343.0
4PO5	1.06E+06	0.92	0.200	2049	394.0
4PO	4.15E+06	0.94	0.080	4682	553.1
4PM	2.90E+06	0.60	0.330	856	1094.8
4PC	5.60E+05	0.92	0.050	1410	361.5
4L35	1.75E+06	0.79	0.220	4457	573.3
4L75	3.80E+06	0.79	0.210	6804	707.2
5PAO	1.88E+05	0.96	0.045	632	247.5
5PAR	9.00E+05	0.95	0.060	1470	395.5
5PC1	1.60E+06	0.91	0.150	2352	540.0
5PO	4.75E+06	0.84	0.160	4509	500.1
5PM	4.70E+05	0.91	0.066	1341	285.9
5L35	1.21E+05	0.90	0.100	2368	443.9
5L75	1.23E+06	0.87	0.130	2529	447.1
7PAO	2.50E+05	0.99	0.027	734	250.8
7PAR	7.10E+05	0.98	0.023	1742	398.5
7PC1	5.40E+05	0.96	0.064	846	346.0
7PO5	1.45E+06	0.87	0.100	2365	414.0
7PO	5.25E+06	0.78	0.230	6383	618.7
7L35	4.90E+05	0.89	0.070	1431	388.9
7L75	1.44E+06	0.89	0.110	2324	445.4
8PAO	1.86E+05	1.00	0.034	670	253.0
8PAR	7.15E+05	0.98	0.019	1832	404.7
8PC1	7.00E+05	0.98	0.096	1276	380.0
8PO5	1.10E+06	0.93	0.080	2430	429.0
8PO	4.20E+06	0.90	0.100	5080	550.4
8L35	1.40E+06	0.86	0.160	2161	436.2
8L75	1.40E+06	0.89	0.090	3484	500.8
(AC-10)					
1PAO	9.10E+05	0.95	0.045	1576	368.7
1PAR	3.50E+06	0.90	0.100	3722	515.3
1PC1	3.20E+06	0.85	0.108	4015	581.0
1PO5	6.50E+06	0.84	0.160	5603	552.0
1PO	1.55E+07	0.74	0.260	13210	788.4
1PM	2.80E+06	0.82	0.200	6235	664.8
1L35	1.67E+07	0.57	0.420	51768	1431.3
1L75	1.12E+07	0.65	0.390	32534	1185.6



Table 3. (continued)

sample ID	VIS 25 poise	C.Flow	S.Index	VIS 60 poise	VIS 135 cSt
11PAO	3.95E+05	0.96	0.037	1110	444.4
11PAR	1.90E+06	0.92	0.070	3558	559.0
11PC1	1.09E+06	0.93	0.077	2024	452.0
11PO5	2.57E+06	0.89	0.140	4602	592.0
11PO	9.50E+06	0.82	0.190	10426	770.3
11L35	3.30E+06	0.83	0.180	4481	638.5
11L75	2.70E+06	0.92	0.090	4220	625.3
(AC-20)					
2PAO	3.40E+06	0.94	0.063	3571	889.2
2PAR	8.20E+06	0.72	0.320	6306	986.7
2PC1	4.30E+06	0.71	0.290	5491	817.0
2PO5	8.00E+06	0.65	0.290	10977	1080.0
2PO	1.95E+07	0.55	0.460	39716	1654.7
2PM	8.30E+06	0.62	0.350	52329	1975.4
2PC	1.20E+07	0.39	0.580	16986	1384.5
2LM	6.50E+06	0.55	0.440	12315	934.1
2L35	1.40E+07	0.58	0.420	55202	1757.9
2L75	1.15E+07	0.66	0.340	17653	1063.9
3PAO	1.17E+06	0.96	0.030	2730	477.3
3PAR	3.60E+06	0.90	0.100	6107	713.3
3PC1	1.45E+06	0.88	0.110	2485	495.0
3PO5	5.70E+06	0.84	0.140	8948	810.0
3PO	1.94E+07	0.72	0.280	21408	1201.6
3PM	4.60E+06	0.81	0.190	15891	1088.2
3PC	6.70E+06	0.72	0.270	13398	964.6
3L35	1.05E+07	0.74	0.260	17218	1039.9
3L75	1.25E+07	0.72	0.290	22750	1183.6
10PAO	1.01E+06	0.97	0.030	2105	459.8
10PAR	3.75E+06	0.93	0.070	6334	732.7
10PC1	7.10E+06	0.78	0.254	14554	1030.0
10PO5	6.00E+06	0.90	0.170	7977	799.0
10PO	1.85E+07	0.73	0.270	18360	1091.0
10L35	1.20E+07	0.69	0.360	37654	1630.2
10L75	1.53E+06	0.90	0.100	2507	489.5
12PAO	1.04E+06	0.98	0.023	2337	470.0
12PAR	4.40E+06	0.91	0.080	6503	774.7
12PC1	2.70E+06	0.91	0.130	2544	440.0
12PO5	6.50E+06	0.90	0.160	9543	828.0
12PO	2.05E+07	0.73	0.260	22624	1139.7
12L35	5.70E+06	0.79	0.200	722	819.7
12L50	4.00E+06	0.89	0.120	4611	715.7
12L75	4.90E+06	0.90	0.100	5489	713.3

VIS 25: viscosity @ 25C; C.Flow: complex flow; S.Index: shear index; VIS 60: viscosity @ 60C; VIS 135: viscosity @ 135C

Table 4. Temperature susceptibility.

sample ID	PR	PI	CN	VTs	PVN,60	PVN,135
(AC-5)						
4PAO	0.354	0.150	7.963	3.381	-0.367	-0.243
4PAR	0.520	-0.632	2.628	3.474	-0.279	-0.347
4PC1	0.357	-0.980	-0.714	3.573	-0.210	-0.481
4PO5	0.360	-0.114	0.552	3.526	-0.247	-0.418
4PO	0.481	-0.153	-2.208	3.576	-0.203	-0.466
4PM	0.389	-0.042	27.546	2.384	-0.329	1.907
4PC	0.429	-0.166	3.981	3.445	-0.316	-0.320
4L35	0.514	1.903	-15.241	3.530	0.937	0.396
4L75	0.527	0.212	-8.185	3.527	0.252	-0.070
5PAO	0.356	0.400	4.636	3.427	-0.171	-0.190
5PAR	0.388	0.221	5.037	3.387	-0.303	-0.204
5PC1	0.398	-0.095	3.844	3.328	-0.160	0.011
5PO	0.472	-0.106	-3.780	3.641	-0.211	-0.585
5PM	0.391	0.983	-7.158	3.624	0.336	-0.211
5L35	0.506	0.153	1.697	3.487	-0.275	-0.364
5L75	0.430	0.214	-2.340	3.507	-0.025	-0.229
7PAO	0.311	-0.640	0.980	3.481	0.030	-0.153
7PAR	0.404	-1.461	2.912	3.451	-0.273	-0.300
7PC1	0.390	-0.558	16.607	3.265	-0.872	-0.388
7PO5	0.381	-0.046	-1.611	3.543	-0.134	-0.370
7PO	0.522	-0.001	-4.995	3.605	-0.089	-0.438
7L35	0.419	-0.478	4.975	3.390	-0.300	-0.207
7L75	0.429	0.388	-1.538	3.477	-0.019	-0.169
8PAO	0.296	-0.803	3.073	3.433	-0.051	-0.116
8PAR	0.411	-0.482	1.684	3.459	-0.201	-0.264
8PC1	0.402	-0.741	7.054	3.362	-0.393	-0.220
8PO5	0.361	0.162	-1.342	3.525	-0.126	-0.330
8PO	0.543	-0.001	-1.390	3.611	-0.308	-0.594
8L35	0.409	0.148	0.636	3.464	-0.153	-0.239
8L75	0.500	0.144	-7.584	3.542	0.273	-0.087
(AC-10)						
1PAO	0.354	-0.620	7.221	3.474	-0.599	-0.569
1PAR	0.420	-0.711	3.467	3.544	-0.485	-0.602
1PC1	0.404	-0.764	2.889	3.480	-0.353	-0.399
1PO5	0.364	-0.519	-2.827	3.645	-0.280	-0.633
1PO	0.519	-0.053	-5.255	3.684	-0.189	-0.626
1PM	0.527	0.434	-7.211	3.542	0.166	-0.155
1L35	0.556	0.818	-28.138	3.718	1.029	0.108
1L75	0.531	0.476	-24.338	3.697	0.875	0.044

Table 4. (continued)

sample ID	PR	PI	CN	VTS	PVN,60	PVN,135
11PAO	0.331	-0.165	8.504	3.175	-0.174	0.307
11PAR	0.420	-0.026	-1.686	3.463	-0.033	-0.151
11PC1	0.396	0.113	2.210	3.409	-0.166	-0.146
11PO5	0.414	-0.080	-3.036	3.517	-0.051	-0.259
11PO	0.600	0.103	-6.383	3.618	-0.034	-0.417
11L35	0.450	0.436	-1.676	3.449	-0.024	-0.117
11L75	0.431	0.419	-2.453	3.442	0.044	-0.057
(AC-20)						
2PAO	0.278	-1.276	13.393	3.116	-0.411	0.226
2PAR	0.447	-0.534	10.235	3.255	-0.382	-0.016
2PC1	0.422	-0.095	5.292	3.341	-0.266	-0.088
2PO5	0.389	-1.278	-1.395	3.392	0.056	0.045
2PO	0.560	0.746	-16.497	3.535	0.676	0.209
2PM	0.514	0.005	-27.443	3.507	1.457	0.790
2PC	0.567	0.066	-2.815	3.372	0.187	0.173
2LM	0.487	0.534	-9.454	3.536	0.283	-0.060
2L35	0.516	0.039	-28.427	3.601	1.306	0.507
2L75	0.457	-0.093	-13.336	3.568	0.453	-0.003
3PAO	0.400	-1.008	0.067	3.485	-0.170	-0.287
3PAR	0.458	-0.230	-1.974	3.481	-0.067	-0.203
3PC1	0.404	-0.484	-0.745	3.419	0.014	-0.036
3PO5	0.390	-0.316	-5.360	3.526	0.059	-0.196
3PO	0.538	0.142	-8.030	3.548	0.187	-0.144
3PM	0.537	0.153	-14.002	3.516	0.601	0.193
3PC	0.600	0.097	-11.564	3.542	0.402	0.008
3L35	0.533	1.038	-9.235	3.575	0.199	-0.185
3L75	0.545	0.357	-16.154	3.579	0.596	0.073
10PAO	0.354	-0.199	3.939	3.411	-0.296	-0.241
10PAR	0.404	-1.211	-1.850	3.474	-0.064	-0.189
10PC1	0.500	0.066	-9.750	3.524	0.317	-0.016
10PO5	0.400	-0.222	-1.967	3.494	-0.086	-0.238
10PO	0.583	-0.100	-3.586	3.563	-0.062	-0.336
10L35	0.594	0.920	-21.449	3.528	1.009	0.446
10L75	0.370	-0.372	0.946	3.431	-0.133	-0.163
12PAO	0.341	-0.764	1.676	3.436	-0.186	-0.209
12PAR	0.426	-0.505	-1.415	3.443	-0.038	-0.114
12PC1	0.403	-0.592	2.979	3.522	-0.421	-0.526
12PO5	0.375	-0.286	-5.961	3.533	0.082	-0.192
12PO	0.609	-0.092	-7.215	3.603	0.062	-0.322
12L35	0.389	0.388	58.905	2.518	-1.981	0.114
12L50	0.415	0.181	-2.116	3.373	0.133	0.134
12L75	0.471	-0.200	-0.724	3.441	-0.077	-0.139

PR: pen. ratio, P4/P25; PI: pen. index; CN: class number;  
VTS: viscosity-temp susceptibility; PVN,60: pen-viscosity number  
@ 60C; PVN,135: pen-viscosity number @ 135C

Table 5. Low-temperature cracking properties.

sample ID	CT C	TES C	S,-23 ksi	S,-29 ksi
(AC-5)				
4PAO	-43.5	-49.0	0.189	0.508
4PAR	-44.0	-38.0	1.740	3.625
4PC1	-38.0	-35.4	2.030	4.930
4PO5	-39.0	-39.2	1.595	3.190
4PO	-43.0	-33.5	4.350	9.425
4PM	-47.0	-44.5	0.399	0.870
4PC	-44.0	-40.5	1.088	2.175
4L35	-48.0	-55.0	0.363	0.580
4L75	-46.0	-36.0	2.900	5.438
5PAO	-42.5	-51.5	0.109	0.363
5PAR	-41.5	-43.0	0.943	1.813
5PC1	-39.5	-38.8	1.740	3.190
5PO	-44.0	-33.5	4.060	7.975
5PM	-45.0	-54.5	0.247	0.363
5L35	-41.0	-39.5	1.450	2.900
5L75	-43.5	-41.0	1.088	2.320
7PAO	-39.0	-44.0	0.218	0.725
7PAR	-39.0	-32.0	3.625	8.700
7PC1	-43.0	-38.8	1.450	2.900
7PO5	-39.5	-39.7	1.305	2.900
7PO	-45.0	-32.5	5.075	10.150
7L35	-45.5	-39.0	1.305	2.900
7L75	-41.5	-43.0	1.088	1.885
8PAO	-40.0	-43.5	0.363	1.160
8PAR	-42.5	-38.0	1.160	2.900
8PC1	-44.0	-37.5	1.450	3.335
8PO5	-39.5	-39.8	1.088	2.465
8PO	-45.0	-32.5	4.785	8.700
8L35	-46.5	-40.5	1.088	2.320
8L75	-46.5	-40.0	1.160	2.610
(AC-10)				
1PAO	-35.0	-36.0	2.610	5.800
1PAR	-36.0	-30.5	6.090	12.325
1PC1	-36.3	-29.1	5.075	11.600
1PO5	-35.0	-28.9	7.250	13.775
1PO	-40.0	-26.0	13.050	21.750
1PM	-42.5	-37.0	2.900	5.800
1L35	-47.5	-49.5	7.250	12.325
1L75	-47.5	-48.5	7.250	11.600

Table 5. (continued)

sample ID	CT C	TES C	S, -23 ksi	S, -29 ksi
11PAO	-42.5	-43.0	0.508	1.450
11PAR	-40.0	-37.0	2.175	4.350
11PC1	-38.5	-41.0	1.305	2.755
11PO5	-37.5	-34.9	3.190	6.670
11PO	-40.0	-29.5	6.525	13.050
11L35	-41.5	-38.0	2.320	5.075
11L75	-42.5	-39.0	2.175	3.915
(AC-20)				
2PAO	-36.0	-28.0	10.875	24.650
2PAR	-35.5	-28.5	10.875	21.750
2PC1	-37.5	-32.2	5.800	10.875
2PO5	-33.0	-24.4	18.850	50.750
2PO	-40.0	-30.0	8.700	14.500
2PM	-40.0	-29.5	7.250	14.500
2PC	-37.5	-28.0	9.425	21.750
2LM	-38.5	-34.0	5.075	8.700
2L35	-34.0	-28.5	10.150	18.850
2L75	-40.0	-31.0	8.700	14.500
3PAO	-37.5	-33.0	4.350	9.425
3PAR	-39.0	-32.0	5.075	10.875
3PC1	-39.0	-36.9	1.450	3.915
3PO5	-32.5	-29.3	7.250	14.500
3PO	-40.0	-27.0	11.600	20.300
3PM	-44.0	-32.0	5.075	10.150
3PC	-44.0	-31.5	5.365	10.440
3L35	-42.5	-34.0	5.800	10.875
3L75	-37.0	-31.0	7.250	13.050
10PAO	-37.0	-38.0	1.740	3.770
10PAR	-37.0	-27.5	9.425	21.750
10PC1	-36.3	-30.5	6.815	12.325
10PO5	-32.5	-29.6	7.250	13.775
10PO	-36.5	-25.0	14.500	24.650
10L35	-45.0	-34.0	5.220	8.700
10L75	-38.5	-37.0	2.320	5.365
12PAO	-35.0	-35.0	2.900	5.510
12PAR	-34.0	-31.0	5.510	10.875
12PC1	-38.5	-33.4	3.625	8.700
12PO5	-32.5	-28.9	7.250	13.775
12PO	-32.5	-24.5	15.950	24.650
12L35	-42.5	-37.0	2.900	5.510
12L50	-41.0	-37.5	2.175	4.350
12L75	-41.5	-33.0	4.350	7.975

CT: cracking temp.; TES: temp. of equivalent stiffness @ 20ksi, 10,000sec; S, -23: stiffness @ -23C, 10,000sec; S, -29: stiffness @ -29C, 20,000sec

Table 6. Viscoelastic properties of thermal cycled samples at +5°C.  
 n : Viscosity, MP  
 G : Elastic shear modulus, psi

Sample	3rd day value after cooling from +25°C		% variation in the first 3 days after				% change*	
			cooling from +25 C		warming from -30 C			
	n	G	n	G	n	G	n	G
J05-01-0	225	12	31	0	29	-38	4.5	-27
J10-01-0	1580	16	44	-18	--	--	--	--
J20-01-0	6990	13	93	-38	--	--	--	--
SC-S	29200	130	71	0	160	650	-140	-118
WR-S	9720	54	43	20	16	-14	13	-25

\*: % difference between third day value after warming from -30°C, and that after cooling from +25°C.

Table 7. HP-GPC results - 3-slice and 4-slice methods.

sample ID	LMS	MMS1	MMS2	SMS	LMS+MMS1
(AC-5s)					
4PAO	4.37	28.21	57.62	9.80	32.58
4PAR	6.27	29.75	54.46	9.52	36.02
4PC1	5.81	30.67	55.57	7.95	36.49
4PO5	7.52	29.91	53.51	9.07	37.42
4PO	7.65	30.54	52.74	9.07	38.19
4PM	4.81	30.46	55.04	9.69	35.28
4PC	3.97	28.74	57.48	9.82	32.71
4L35	5.27	30.60	54.96	9.18	35.86
4L75	6.10	31.47	53.71	8.71	37.57
5PAO	4.07	27.73	58.04	10.16	31.80
5PAR	6.30	29.62	54.46	9.62	35.92
5PC1	4.82	31.62	56.37	7.19	36.45
5PO	7.80	30.74	52.46	9.00	38.54
5PM	3.94	29.78	56.92	9.36	33.72
5L35	5.90	30.83	54.41	8.87	36.73
7PAO	1.16	22.83	62.75	13.26	23.99
7PAR	5.72	29.34	55.16	9.79	35.06
7PC1	4.09	29.43	58.67	7.83	33.52
7PO5	6.43	29.82	54.34	9.41	36.25
7PO	7.55	30.81	52.48	9.16	38.36
7L35	5.14	29.82	55.58	9.47	34.96
8PAO	3.84	28.38	57.93	9.86	32.22
8PAR	5.96	29.62	54.77	9.66	35.57
8PC1	5.91	31.57	54.36	8.16	37.48
8PO5	6.39	29.71	54.53	9.38	36.09
8PO	6.85	30.42	53.53	9.20	37.27
8L35	4.94	29.97	55.63	9.46	34.91
(AC-10s)					
1PAO	3.35	25.69	61.21	9.74	29.05
1PAR	4.53	27.49	58.48	9.51	32.01
1PC1	4.37	29.76	57.16	8.70	34.13
1PO5	5.02	27.75	57.79	9.45	32.77
1PO	5.60	29.07	56.63	8.70	34.67
1PM	3.62	28.80	57.98	9.60	32.42
1L35	4.43	29.45	56.98	9.13	33.89
1L75	3.05	28.40	58.74	9.81	31.45

Table 7. (continued)

sample ID	LMS	MMS1	MMS2	SMS	LMS+MMS1
11PAO	3.78	28.53	57.97	9.72	32.31
11PAR	6.07	30.23	54.48	9.23	36.30
11PC1	6.73	30.16	53.96	9.16	36.88
11PO5	6.71	30.09	54.08	9.13	36.79
11PO	7.07	30.98	52.97	8.98	38.05
11L35	5.18	29.84	55.31	9.67	35.02
(AC-20s)					
2PAO	4.56	31.34	56.89	7.21	35.90
2PAR	6.17	33.46	53.48	6.90	39.63
2PC1	6.23	33.06	52.96	7.73	39.30
2PO5	6.54	33.48	52.89	7.09	40.02
2PO	7.45	35.22	50.98	6.36	42.66
2PM	5.72	34.48	52.86	6.95	40.19
2PC	5.83	34.44	52.79	6.94	40.27
2LM	5.91	33.08	53.31	7.70	38.99
2L35	5.99	33.69	53.04	7.29	39.68
2L75	5.77	33.10	53.48	7.66	38.87
3PAO	4.44	29.25	57.40	8.91	33.68
3PAR	6.59	30.77	54.15	8.49	37.36
3PC1	6.29	30.43	54.74	8.55	36.72
3PO5	7.33	30.56	53.50	8.61	37.89
3PO	7.98	31.86	52.24	7.92	39.84
3PM	4.61	31.16	55.05	9.19	35.77
3PC	4.30	30.81	56.13	8.76	35.11
3L35	5.72	31.77	54.17	8.35	37.49
3L75	5.96	32.19	53.60	8.26	38.15
10PAO	3.93	28.75	57.96	9.37	32.68
10PAR	6.27	30.48	54.39	8.87	36.76
10PC1	4.95	31.40	54.86	8.79	36.35
10PO5	6.82	30.27	54.10	8.82	37.09
10PO	7.34	31.11	52.95	8.60	38.45
10L35	11.28	31.66	49.35	7.70	42.94
12PAO	4.33	29.53	57.06	9.09	33.86
12PAR	6.54	30.75	54.04	8.67	37.29
12PC1	5.38	29.21	56.35	9.06	34.59
12PO5	6.93	30.53	53.91	8.62	37.46
12PO	7.49	31.55	52.55	8.42	39.03
12L35	5.58	30.63	54.71	9.08	36.21



Table 8. HP-GPC results - 8-slice method.

sample ID	X1	X2	X3	X4	X5	X6	X7	X8
(AC-5s)								
4PAO	13.80	14.68	17.24	13.29	12.21	12.67	7.87	8.25
4PAR	17.14	14.88	16.49	12.56	11.50	11.91	7.50	8.02
4PC1	16.69	15.65	17.09	13.11	11.84	12.04	6.96	6.63
4PO5	18.71	14.74	16.34	12.30	11.37	11.64	7.27	7.63
4PO	19.20	15.07	16.11	12.17	11.04	11.55	7.24	7.62
4PM	15.13	15.70	17.83	12.73	11.33	11.70	7.39	8.20
4PC	13.45	15.04	17.56	13.31	12.04	12.49	7.87	8.24
4L35	16.17	15.52	17.30	12.77	11.49	11.74	7.28	7.74
4L75	17.57	15.78	17.22	12.57	11.16	11.37	7.02	7.32
5PAO	13.30	14.42	17.13	13.40	12.30	12.83	8.06	8.56
5PAR	17.12	14.82	16.44	12.55	11.44	11.98	7.53	8.11
5PC1	15.77	16.30	18.22	13.85	12.04	11.50	6.36	5.97
5PO	19.56	15.04	16.07	12.10	10.97	11.50	7.18	7.57
5PM	13.65	15.63	18.09	13.22	11.82	12.16	7.59	7.85
5L35	17.10	15.50	16.91	12.64	11.37	11.76	7.32	7.41
7PAO	7.39	12.78	16.50	13.77	13.21	14.96	10.21	11.19
7PAR	16.20	14.83	16.67	12.76	11.61	12.10	7.58	8.27
7PC1	13.81	15.40	18.07	14.30	12.65	12.33	6.95	6.50
7PO5	17.11	15.04	16.83	12.60	11.48	11.67	7.34	7.94
7PO	19.29	15.11	16.16	12.12	11.01	11.42	7.15	7.73
7L35	15.66	15.17	17.08	12.89	11.72	12.06	7.45	7.99
8PAO	13.24	14.83	17.40	13.43	12.26	12.69	7.85	8.31
8PAR	16.63	14.93	16.58	12.68	11.51	12.00	7.52	8.16
8PC1	17.37	15.95	17.01	12.80	11.40	11.68	7.01	6.78
8PO5	17.02	15.02	16.78	12.63	11.44	11.86	7.37	7.90
8PO	18.27	15.04	16.33	12.38	11.25	11.69	7.29	7.75
8L35	15.41	15.33	17.21	12.90	11.65	12.07	7.47	7.97
(AC-10s)								
1PAO	11.65	13.44	17.23	14.34	13.29	13.73	8.16	8.16
1PAR	13.88	14.15	16.87	13.65	12.69	13.01	7.76	7.99
1PC1	14.24	15.66	17.65	13.44	12.19	12.26	7.28	7.27
1PO5	14.44	14.32	17.05	13.53	12.48	12.70	7.52	7.97
1PO	16.04	14.66	16.69	13.19	12.28	12.57	7.23	7.34
1PM	12.97	15.10	17.90	13.42	12.20	12.58	7.78	8.06
1L35	14.52	15.22	17.43	13.30	12.11	12.33	7.41	7.69
1L75	12.23	14.97	17.74	13.54	12.45	12.93	7.89	8.26

Table 8. (continued)

sample ID	X1	X2	X3	X4	X5	X6	X7	X8
11PAO	13.25	14.88	17.55	13.50	12.33	12.55	7.75	8.19
11PAR	17.02	15.19	16.80	12.70	11.46	11.77	7.28	7.78
11PC1	17.72	15.09	16.64	12.56	11.42	11.65	7.17	7.75
11PO5	17.50	15.23	16.75	12.74	11.29	11.64	7.14	7.71
11PO	18.71	15.31	16.46	12.32	11.13	11.43	7.06	7.58
11L35	15.70	15.24	17.03	12.95	11.52	11.97	7.39	8.20
(AC-20s)								
2PAO	14.87	16.42	19.25	14.49	12.05	10.93	5.92	6.08
2PAR	18.02	17.04	18.60	13.53	11.25	10.22	5.50	5.85
2PC1	17.95	16.85	18.19	13.32	11.08	10.26	5.74	6.61
2PO5	18.22	17.20	18.82	13.35	11.07	9.91	5.39	6.05
2PO	20.53	17.61	18.06	12.84	10.69	9.78	5.09	5.41
2PM	17.82	17.65	18.90	13.41	10.96	9.96	5.37	5.92
2PC	18.24	17.42	18.79	13.32	10.99	9.93	5.42	5.90
2LM	17.75	16.79	18.03	13.08	11.20	10.58	6.03	6.53
2L35	18.20	17.03	18.18	13.05	11.09	10.44	5.87	6.16
2L75	17.69	16.76	18.03	13.09	11.27	10.62	6.05	6.49
3PAO	14.32	15.13	17.70	13.54	12.28	12.26	7.27	7.50
3PAR	17.85	15.44	16.90	12.80	11.59	11.45	6.82	7.15
3PC1	17.17	15.36	17.08	12.85	11.67	11.74	6.94	7.21
3PO5	18.36	15.38	16.86	12.67	11.39	11.30	6.76	7.28
3PO	20.15	15.68	16.44	12.30	11.15	11.18	6.40	6.71
3PM	15.04	16.16	18.24	12.92	11.43	11.48	6.92	7.82
3PC	14.43	16.06	18.52	13.21	11.67	11.71	7.03	7.37
3L35	17.19	16.02	17.49	12.90	11.41	11.28	6.66	7.05
3L75	17.60	16.24	17.60	12.80	11.21	11.06	6.54	6.96
10PAO	13.46	15.04	17.63	13.65	12.40	12.47	7.46	7.90
10PAR	17.35	15.31	16.92	12.77	11.51	11.64	7.02	7.49
10PC1	16.08	16.00	17.43	12.83	11.58	11.69	6.98	7.41
10PO5	17.75	15.20	17.09	12.70	11.45	11.47	6.90	7.45
10PO	19.10	15.32	16.53	12.43	11.23	11.32	6.82	7.26
10L35	23.78	15.27	15.80	11.60	10.37	10.46	6.27	6.46
12PAO	14.34	15.29	17.70	13.42	12.12	12.20	7.27	7.66
12PAR	17.76	15.40	16.94	12.74	11.45	11.51	6.89	7.32
12PC1	15.55	14.98	16.83	13.16	12.02	12.38	7.50	7.59
12PO5	17.91	15.43	17.05	12.83	11.31	11.36	6.83	7.27
12PO	19.42	15.54	16.58	12.40	11.10	11.16	6.69	7.11
12L35	16.57	15.47	17.24	12.80	11.50	11.64	7.08	7.68

Table 9. HP-GPC results -  
molecular weight distribution characteristics.

sample ID	WTMWT	ZMWT	Z1MWT	POLYIDX
(AC-5s)				
4PAO	6.63E+03	4.11E+04	8.05E+04	11.484
4PAR	8.35E+03	4.86E+04	8.87E+04	13.857
4PC1	8.12E+03	4.76E+04	9.07E+04	12.472
4PO5	9.45E+03	5.43E+04	9.50E+04	15.183
4PO	9.61E+03	5.41E+04	9.59E+04	15.318
4PM	7.23E+03	4.37E+04	9.13E+04	12.073
4PC	6.42E+03	4.09E+04	9.01E+04	11.061
4L35	7.53E+03	4.16E+04	7.70E+04	12.270
4L75	8.35E+03	4.64E+04	8.81E+04	13.080
5PAO	6.33E+03	3.90E+04	7.60E+04	11.211
5PAR	8.35E+03	4.86E+04	8.79E+04	13.934
5PC1	7.37E+03	4.09E+04	7.97E+04	10.746
5PO	9.76E+03	5.45E+04	9.73E+04	15.466
5PM	6.50E+03	4.11E+04	9.49E+04	10.859
5L35	8.14E+03	4.63E+04	8.85E+04	12.989
7PAO	3.54E+03	1.94E+04	3.86E+04	7.596
7PAR	7.88E+03	4.76E+04	9.04E+04	13.359
7PC1	6.57E+03	3.93E+04	7.99E+04	10.230
7PO5	8.56E+03	5.13E+04	9.40E+04	14.070
7PO	9.50E+03	5.22E+04	9.16E+04	15.184
7L35	7.37E+03	4.21E+04	7.85E+04	12.298
8PAO	6.21E+03	3.74E+04	7.60E+04	10.813
8PAR	8.08E+03	4.76E+04	8.84E+04	13.553
8PC1	8.27E+03	4.65E+04	8.88E+04	12.671
8PO5	8.53E+03	5.14E+04	9.38E+04	14.036
8PO	8.87E+03	4.96E+04	8.90E+04	14.359
8L35	7.23E+03	4.13E+04	7.80E+04	12.055
(AC-10s)				
1PAO	5.57E+03	3.57E+04	7.10E+04	9.962
1PAR	6.70E+03	4.21E+04	8.10E+04	11.562
1PC1	6.79E+03	4.03E+04	7.96E+04	11.000
1PO5	7.21E+03	4.71E+04	9.03E+04	12.284
1PO	7.70E+03	4.50E+04	8.27E+04	12.537
1PM	6.10E+03	3.76E+04	8.33E+04	10.456
1L35	6.76E+03	3.85E+04	7.28E+04	11.234
1L75	5.54E+03	3.03E+04	5.88E+04	9.698

Table 9. (continued)

sample ID	WTMWT	ZMWT	Z1MWT	POLYIDX
11PAO	6.15E+03	3.58E+04	7.00E+04	10.625
11PAR	8.22E+03	4.72E+04	8.69E+04	13.410
11PC1	8.85E+03	5.22E+04	9.48E+04	14.320
11PO5	8.77E+03	5.12E+04	9.16E+04	14.152
11PO	9.15E+03	5.10E+04	9.17E+04	14.525
11L35	7.37E+03	4.16E+04	7.72E+04	12.395
(AC-20s)				
2PAO	7.21E+03	4.32E+04	9.00E+04	10.444
2PAR	8.74E+03	4.84E+04	9.39E+04	11.999
2PC1	8.87E+03	5.20E+04	1.04E+05	12.789
2PO5	9.17E+03	5.29E+04	1.02E+05	12.615
2PO	9.95E+03	5.13E+04	9.51E+04	12.890
2PM	8.38E+03	4.43E+04	8.80E+04	11.440
2PC	8.54E+03	4.54E+04	9.05E+04	11.640
2LM	8.47E+03	4.76E+04	9.67E+04	12.304
2L35	8.50E+03	4.41E+04	8.26E+04	11.988
2L75	8.33E+03	4.58E+04	9.20E+04	12.096
3PAO	6.77E+03	3.97E+04	7.61E+04	11.126
3PAR	8.74E+03	4.97E+04	9.13E+04	13.591
3PC1	8.53E+03	5.07E+04	9.38E+04	13.424
3PO5	9.44E+03	5.54E+04	9.87E+04	14.696
3PO	1.00E+04	5.39E+04	9.54E+04	14.840
3PM	7.13E+03	4.23E+04	9.03E+04	11.575
3PC	6.86E+03	4.13E+04	9.07E+04	10.960
3L35	8.03E+03	4.31E+04	7.95E+04	12.350
3L75	8.33E+03	4.59E+04	8.84E+04	12.623
10PAO	6.31E+03	3.73E+04	7.45E+04	10.686
10PAR	8.44E+03	4.85E+04	8.99E+04	13.464
10PC1	7.40E+03	4.02E+04	7.55E+04	11.775
10PO5	8.96E+03	5.31E+04	9.67E+04	14.176
10PO	9.37E+03	5.16E+04	9.18E+04	14.546
10L35	1.38E+04	7.76E+04	1.30E+05	19.563
12PAO	6.69E+03	3.83E+04	7.40E+04	11.051
12PAR	8.70E+03	4.98E+04	9.13E+04	13.677
12PC1	7.57E+03	4.56E+04	8.54E+04	12.490
12PO5	9.05E+03	5.26E+04	9.40E+04	14.124
12PO	9.52E+03	5.16E+04	9.14E+04	14.555
12L35	7.81E+03	4.34E+04	7.98E+04	12.625

WTMWT : weighted average molecular weight.  
 ZMWT : Z average molecular weight.  
 Z1MWT : Z+1 average molecular weight.  
 POLYIDX : polydisperse index.

Table 10. HP-GPC results - percent changes,  
3-slice and 4-slice methods.

sample ID	LMS	MMS1	MMS2	SMS	LMS+MMS1
(AC-5s)					
4PAO	0.00	0.00	0.00	0.00	0.00
4PAR	43.35	5.47	-5.48	-2.91	10.55
4PC1	32.90	8.75	-3.56	-18.92	11.99
4PO5	71.81	6.03	-7.14	-7.44	14.86
4PO	74.83	8.28	-8.48	-7.47	17.21
4PM	10.06	8.00	-4.48	-1.16	8.28
4PC	-9.35	1.89	-0.25	0.16	0.38
4L35	20.39	8.47	-4.61	-6.37	10.07
4L75	39.48	11.58	-6.78	-11.11	15.32
5PAO	0.00	0.00	0.00	0.00	0.00
5PAR	54.60	6.85	-6.16	-5.36	12.97
5PC1	18.36	14.06	-2.88	-29.27	14.61
5PO	91.55	10.86	-9.62	-11.41	21.20
5PM	-3.19	7.40	-1.93	-7.87	6.04
5L35	44.86	11.20	-6.26	-12.74	15.51
7PAO	0.00	0.00	0.00	0.00	0.00
7PAR	391.15	28.54	-12.10	-26.21	46.13
7PC1	251.20	28.92	-6.50	-41.01	39.70
7PO5	452.15	30.65	-13.40	-29.04	51.10
7PO	548.54	34.98	-16.37	-30.93	59.90
7L35	341.24	30.63	-11.43	-28.64	45.70
8PAO	0.00	0.00	0.00	0.00	0.00
8PAR	55.12	4.34	-5.45	-2.01	10.39
8PC1	53.97	11.24	-6.16	-17.21	16.33
8PO5	66.35	4.66	-5.86	-4.88	12.01
8PO	78.54	7.17	-7.59	-6.68	15.67
8L35	28.71	5.60	-3.96	-4.08	8.35
(AC-10s)					
1PAO	0.00	0.00	0.00	0.00	0.00
1PAR	34.94	6.98	-4.46	-2.39	10.21
1PC1	30.32	15.84	-6.62	-10.70	17.51
1PO5	49.61	8.00	-5.59	-3.03	12.81
1PO	66.88	13.16	-7.49	-10.65	19.36
1PM	8.05	12.10	-5.28	-1.49	11.63
1L35	32.20	14.63	-6.91	-6.26	16.66
1L75	-9.21	10.55	-4.04	0.73	8.27

Table 10. (continued)

sample ID	LMS	MMS1	MMS2	SMS	LMS+MMS1
11PAO	0.00	0.00	0.00	0.00	0.00
11PAR	60.65	5.94	-6.02	-5.09	12.34
11PC1	77.98	5.70	-6.92	-5.77	14.15
11PO5	77.48	5.46	-6.70	-6.14	13.88
11PO	87.19	8.58	-8.62	-7.64	17.77
11L35	37.05	4.60	-4.58	-0.50	8.39
(AC-20s)					
2PAO	0.00	0.00	0.00	0.00	0.00
2PAR	35.25	6.75	-6.00	-4.29	10.37
2PC1	36.52	5.50	-6.91	7.28	9.44
2PO5	43.36	6.82	-7.03	-1.61	11.46
2PO	63.19	12.36	-10.39	-11.82	18.83
2PM	25.22	10.01	-7.09	-3.54	11.95
2PC	27.67	9.89	-7.21	-3.75	12.15
2LM	29.45	5.55	-6.29	6.81	8.58
2L35	31.27	7.49	-6.78	1.17	10.51
2L75	26.40	5.60	-6.01	6.27	8.25
3PAO	0.00	0.00	0.00	0.00	0.00
3PAR	48.60	5.21	-5.66	-4.77	10.93
3PC1	41.79	4.03	-4.64	-4.08	9.00
3PO5	65.26	4.50	-6.79	-3.43	12.50
3PO	79.91	8.93	-8.99	-11.11	18.28
3PM	3.92	6.53	-4.09	3.05	6.19
3PC	-3.00	5.35	-2.21	-1.78	4.25
3L35	29.01	8.62	-5.62	-6.36	11.31
3L75	34.24	10.07	-6.62	-7.38	13.25
10PAO	0.00	0.00	0.00	0.00	0.00
10PAR	59.50	6.05	-6.16	-5.36	12.48
10PC1	25.91	9.22	-5.34	-6.16	11.23
10PO5	73.30	5.30	-6.65	-5.89	13.49
10PO	86.50	8.23	-8.63	-8.16	17.65
10L35	186.78	10.15	-14.84	-17.83	31.41
12PAO	0.00	0.00	0.00	0.00	0.00
12PAR	51.07	4.12	-5.28	-4.60	10.12
12PC1	24.40	-1.10	-1.23	-0.31	2.16
12PO5	60.23	3.37	-5.51	-5.13	10.63
12PO	73.08	6.81	-7.90	-7.32	15.28
12L35	29.05	3.70	-4.11	-0.07	6.94

Table 11. HP-GPC results - percent changes, 8-slice method.

sample ID	X1	X2	X3	X4	X5	X6	X7	X8
(AC-5s)								
4PAO	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
4PAR	24.19	1.38	-4.33	-5.54	-5.81	-6.01	-4.68	-2.70
4PC1	20.97	6.63	-0.88	-1.39	-3.01	-5.00	-11.54	-19.66
4PO5	35.62	0.42	-5.21	-7.46	-6.92	-8.12	-7.55	-7.48
4PO	39.18	2.65	-6.57	-8.48	-9.57	-8.87	-7.97	-7.57
4PM	9.63	7.00	3.40	-4.27	-7.23	-7.65	-6.10	-0.52
4PC	-2.53	2.47	1.87	0.12	-1.41	-1.40	0.00	-0.05
4L35	17.18	5.72	0.37	-3.91	-5.87	-7.39	-7.46	-6.21
4L75	27.31	7.55	-0.13	-5.46	-8.63	-10.27	-10.78	-11.25
5PAO	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
5PAR	28.71	2.80	-4.02	-6.32	-6.99	-6.58	-6.61	-5.25
5PC1	18.58	13.04	6.35	3.38	-2.19	-10.36	-21.13	-30.24
5PO	47.09	4.33	-6.19	-9.68	-10.82	-10.35	-10.91	-11.57
5PM	2.64	8.36	5.59	-1.39	-3.90	-5.22	-5.87	-8.29
5L35	28.60	7.51	-1.29	-5.69	-7.62	-8.35	-9.18	-13.45
7PAO	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
7PAR	119.28	16.01	1.00	-7.33	-12.15	-19.16	-25.73	-26.05
7PC1	87.00	20.51	9.54	3.86	-4.27	-17.59	-31.97	-41.91
7PO5	131.71	17.67	1.99	-8.50	-13.14	-22.02	-28.14	-29.00
7PO	161.22	18.25	-2.04	-11.97	-16.68	-23.67	-29.96	-30.87
7L35	111.98	18.68	3.49	-6.38	-11.32	-19.41	-27.03	-28.63
8PAO	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
8PAR	25.59	0.68	-4.71	-5.64	-6.13	-5.38	-4.21	-1.86
8PC1	31.15	7.54	-2.20	-4.70	-7.01	-7.92	-10.72	-18.38
8PO5	28.52	1.27	-3.53	-6.02	-6.71	-6.56	-6.13	-4.94
8PO	37.96	1.44	-6.11	-7.87	-8.26	-7.83	-7.11	-6.79
8L35	16.34	3.40	-1.09	-3.97	-4.99	-4.89	-4.75	-4.13
(AC-10s)								
1PAO	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
1PAR	19.15	5.28	-2.12	-4.82	-4.47	-5.26	-4.85	-2.11
1PC1	22.18	16.58	2.42	-6.28	-8.26	-10.71	-10.81	-10.88
1PO5	23.92	6.62	-1.09	-5.68	-6.08	-7.48	-7.87	-2.38
1PO	37.69	9.10	-3.15	-8.06	-7.55	-8.41	-11.39	-10.13
1PM	11.33	12.37	3.86	-6.48	-8.17	-8.36	-4.66	-1.31
1L35	24.61	13.30	1.15	-7.29	-8.87	-10.18	-9.23	-5.84
1L75	4.93	11.43	2.93	-5.61	-6.33	-5.81	-3.30	1.19

Table 11. (continued)

sample ID	X1	X2	X3	X4	X5	X6	X7	X8
11PAO	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
11PAR	28.45	2.06	-4.25	-5.93	-7.00	-6.21	-6.13	-4.97
11PC1	33.79	1.38	-5.16	-6.96	-7.34	-7.23	-7.45	-5.43
11PO5	32.10	2.30	-4.52	-5.65	-8.37	-7.27	-7.88	-5.88
11PO	41.20	2.88	-6.21	-8.76	-9.68	-8.92	-8.85	-7.47
11L35	18.51	2.38	-2.92	-4.04	-6.56	-4.64	-4.63	0.12
(AC-20s)								
2PAO	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
2PAR	21.22	3.74	-3.39	-6.64	-6.62	-6.48	-7.00	-3.85
2PC1	20.75	2.62	-5.53	-8.08	-8.06	-6.14	-3.01	8.70
2PO5	22.56	4.71	-2.22	-7.89	-8.15	-9.35	-8.81	-0.56
2PO	38.08	7.20	-6.17	-11.39	-11.32	-10.51	-14.02	-11.03
2PM	19.88	7.50	-1.80	-7.47	-9.00	-8.85	-9.25	-2.60
2PC	22.70	6.08	-2.42	-8.12	-8.81	-9.11	-8.40	-3.01
2LM	19.43	2.25	-6.32	-9.72	-7.09	-3.17	1.94	7.30
2L35	22.45	3.68	-5.57	-9.98	-7.98	-4.46	-0.85	1.30
2L75	19.02	2.05	-6.33	-9.67	-6.50	-2.80	2.30	6.66
3PAO	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
3PAR	24.65	2.02	-4.48	-5.42	-5.63	-6.67	-6.11	-4.65
3PC1	19.90	1.51	-3.50	-5.10	-4.99	-4.28	-4.57	-3.89
3PO5	28.22	1.67	-4.71	-6.41	-7.25	-7.88	-6.99	-2.95
3PO	40.71	3.63	-7.13	-9.12	-9.26	-8.82	-11.90	-10.55
3PM	5.04	6.77	3.09	-4.51	-6.96	-6.41	-4.80	4.23
3PC	0.78	6.16	4.63	-2.40	-4.96	-4.54	-3.27	-1.73
3L35	20.07	5.87	-1.15	-4.70	-7.08	-8.03	-8.31	-6.00
3L75	22.89	7.35	-0.56	-5.46	-8.72	-9.86	-10.04	-7.15
10PAO	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
10PAR	28.94	1.82	-4.04	-6.44	-7.17	-6.62	-5.88	-5.29
10PC1	19.49	6.43	-1.16	-5.99	-6.62	-6.23	-6.47	-6.25
10PO5	31.92	1.07	-3.08	-6.97	-7.63	-7.97	-7.59	-5.76
10PO	41.98	1.89	-6.28	-8.99	-9.43	-9.22	-8.60	-8.09
10L35	76.75	1.54	-10.42	-15.07	-16.37	-16.10	-16.00	-18.26
12PAO	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
12PAR	23.86	0.77	-4.31	-5.10	-5.57	-5.66	-5.26	-4.53
12PC1	8.42	-2.01	-4.94	-1.95	-0.88	1.47	3.19	-0.91
12PO5	24.93	0.96	-3.71	-4.39	-6.71	-6.89	-6.04	-5.11
12PO	35.46	1.69	-6.36	-7.62	-8.44	-8.48	-7.95	-7.22
12L35	15.55	1.22	-2.60	-4.58	-5.10	-4.56	-2.55	0.23



Table 12. Effects of Oxidative Aging  
on LMS and Log Vis 25

Type	Project	Source	$\Delta$ (LMS, %)	$\Delta$ Log VIS 25	$\Delta$ Log VIS 25
					$\Delta$ (LMS, %)
AC-5	4	Koch, Dubuque	5.61	1.44	0.26
AC-5	5	Koch, Dubuque	6.74	1.40	0.21
AC-5	7	Koch, Algona	14.37	1.32	0.09
AC-5	8	Koch, Algona	5.05	1.35	0.27
AC-10	1	Koch, Algona	5.62	1.23	0.22
AC-10	11	Koch, Algona	5.74	1.38	0.24
AC-20	2	Koch, Tama	6.76	0.76	0.11
AC-20	3	Koch, Dubuque	6.16	1.22	0.20
AC-20	10	Jebro, Sioux City	5.77	1.26	0.22
AC-20	12	Koch, Omaha	5.17	1.29	0.25

Table 13. Summary of TMA results.

sample ID	Tg C	Tsp C	ML um/C	MH um/C
(AC-5s)				
4PAO	-31.3	7.0	0.176	0.466
4PAR	-30.3	5.0	0.189	0.441
4PC1	-37.7	3.9	0.127	0.559
4PO5	-28.8	7.3	0.132	0.422
4PO	-27.3	13.0	0.213	0.532
4PM	-29.5	6.0	0.158	0.441
4PC	-29.3	8.0	0.249	0.512
4L35	-33.5	7.0	0.162	0.547
4L75	-33.3	12.5	0.162	0.487
5PAO	-30.0	14.5	0.149	0.510
5PAR	-36.3	7.0	0.039	0.407
5PC1	-36.7	6.9	0.047	0.433
5PO	-34.0	13.0	0.162	0.503
5PM	-31.3	5.0	0.106	0.451
5L35	-30.5	10.5	0.160	0.561
5L75	-32.5	10.5	0.167	0.510
7PAO	-34.0	15.0	0.195	0.494
7PAR	-26.8	10.5	0.215	0.571
7PC1	-36.5	5.2	0.051	0.380
7PO5	-29.4	7.0	0.193	0.452
7PO	-28.5	14.5	0.231	0.577
7L35	-31.0	3.0	0.249	0.603
7L75	-37.0	12.5	0.225	0.618
8PAO	-26.8	15.0	0.209	0.577
8PAR	-29.9	12.0	0.264	0.695
8PC1	-32.1	4.2	0.079	0.331
8PO5	-29.2	6.8	0.141	0.464
8PO	-30.9	12.5	0.174	0.392
8L35	-32.0	9.5	0.113	0.441
8L75	-33.3	7.0	0.240	0.630
(AC-10s)				
1PAO	-33.0	-4.0	0.094	0.299
1PAR	-22.5	14.0	0.208	0.682
1PC1	-35.3	10.1	0.182	0.374
1PO5	-30.9	12.2	0.105	0.435
1PO	-27.5	12.5	0.264	0.647
1PM	-31.9	12.0	0.235	0.566
1L35	-28.0	25.0	0.249	0.483
1L75	-28.0	14.5	0.216	0.477

Table 13. (continued)

sample ID	Tg C	Tsp C	ML um/C	MH um/C
11PAO	-27.5	3.5	0.126	0.488
11PAR	-28.0	12.0	0.259	0.687
11PC1	-34.9	4.5	0.139	0.454
11PO5	-29.8	9.4	0.182	0.386
11PO	-24.0	13.5	0.180	0.451
11L35	-34.0	4.0	0.251	0.732
11L75	-25.5	25.0	0.141	0.505
(AC-20s)				
2PAO	-25.0	17.5	0.167	0.477
2PAR	-28.5	16.0	0.240	0.508
2PC1	-30.6	14.9	0.213	0.380
2PO5	-29.0	15.5	0.268	0.490
2PO	-28.3	25.0	0.231	0.481
2PM	-27.8	17.5	0.224	0.514
2PC	-32.5	18.0	0.235	0.503
2LM	-29.9	12.0	0.260	0.503
2L35	-33.3	17.5	0.244	0.440
2L75	-33.9	19.5	0.138	0.429
3PAO	-22.5	12.0	0.117	0.499
3PAR	-27.0	7.0	0.154	0.367
3PC1	-34.4	7.0	0.114	0.404
3PO5	-24.5	12.2	0.198	0.431
3PO	-22.0	17.5	0.231	0.545
3PM	-29.4	17.5	0.214	0.510
3PC	-33.0	12.5	0.186	0.465
3L35	-27.5	16.0	0.220	0.521
3L75	-25.3	25.0	0.211	0.507
10PAO	-23.5	13.0	0.204	0.523
10PAR	-22.5	13.5	0.244	0.601
10PC1	-34.9	8.8	0.109	0.375
10PO5	-27.8	9.2	0.109	0.388
10PO	-28.5	14.0	0.212	0.508
10L35	-31.0	15.0	0.245	0.477
10L75	-32.0	19.5	0.160	0.444
12PAO	-24.0	13.0	0.222	0.625
12PAR	-25.0	11.5	0.195	0.521
12PC1	-30.1	11.0	0.123	0.402
12PO5	-23.1	13.4	0.211	0.483
12PO	-21.5	25.0	0.203	0.554
12L35	-28.0	25.0	0.268	0.657
12L50	-27.3	12.5	0.182	0.521
12L75	-28.3	11.5	0.191	0.525

Tg: glass transition temp., Tsp: softening temp., ML & MH: slopes of the expansion curve below and above Tg, respectively

Table 14. Water sensitivity of mixes.

Project	One Year Old Core Samples		
	% Air	RM ratio	ITS ratio
AC-5			
4	5.26	1.05	0.93
5	4.91	0.95	0.95
7	2.81	1.29	1.26
8	2.77	1.38	1.31
AC-10			
1	3.83	1.10	1.16
11	4.04	0.39	0.52
AC-20			
2	6.43	0.96	1.05
3	5.52	1.10	1.09
10	7.19	1.04	0.75
12	5.46	1.26	1.00

Table 15. Predicted rheological properties from lab aging test.

Project	Y5	Y10	Y20	Y30	Ult.Prop.
Penetration at 5 C					
4	10	9.83	9.73	9.70	9.64
5	11	--	--	--	--
7	10	9.71	9.54	9.47	9.33
8	10	9.80	9.69	9.65	9.57
1	6	6.00	6.00	6.00	6.00
11	7	6.80	6.69	6.65	6.57
2	6	5.91	5.91	5.92	5.92
3	6	6.08	6.12	6.13	6.15
10	5	5.00	5.00	5.00	5.00
12	4	3.71	3.54	3.47	3.33
Penetration at 25 C					
4	52	31.08	11.87	2.84	--
5	53	--	--	--	--
7	46	2.57	--	--	--
8	46	15.27	--	--	--
1	27	14.39	0.93	--	--
11	35	22.97	13.07	8.76	--
2	25	--	--	--	--
3	26	17.95	11.22	8.26	0.62
10	24	14.80	6.75	3.09	--
12	23	12.54	2.93	--	--
Penetration at 4 C					
4	25	24.00	23.47	23.29	22.92
5	25	--	--	--	--
7	24	21.20	19.33	18.62	17.00
8	25	23.96	23.37	23.17	22.74
1	14	13.63	13.43	13.36	13.22
11	21	20.35	19.99	19.86	19.59
2	14	14.00	14.00	14.00	14.00
3	14	13.65	13.47	13.40	13.27
10	14	13.55	13.29	13.19	13.00
12	14	13.85	13.77	13.74	13.68
R&B softening point, C					
4	54.0	56.1	57.6	58.2	59.5
5	54.0	--	--	--	--
7	56.0	58.1	59.5	60.0	61.1
8	56.0	58.3	60.0	60.6	62.1
1	61.5	69.0	80.0	87.7	--
11	59.5	66.9	80.2	91.9	--
2	67.0	--	--	--	--
3	63.0	85.4	--	--	--
10	62.5	64.3	65.3	65.7	66.6
12	63.0	67.5	71.9	74.1	80.4

Table 15. (continued)

Project	Y5	Y10	Y20	Y30	Ult.Prop.
Viscosity at 25 C, poise					
4	4.15E+06	--	--	--	--
5	4.75E+06	--	--	--	--
7	5.25E+06	1.34E+07	1.23E+08	--	--
8	4.20E+06	--	--	--	--
1	1.55E+07	2.27E+07	3.09E+07	3.55E+07	5.15E+07
11	9.50E+06	--	--	--	--
2	1.95E+07	--	--	--	--
3	1.94E+07	8.92E+07	--	--	--
10	1.85E+07	5.20E+07	--	--	--
12	2.05E+07	1.01E+08	--	--	--
Viscosity at 60 C, poise					
4	4682	11694	--	--	--
5	4509	--	--	--	--
7	6383	25686	--	--	--
8	5080	9113	21038	44132	--
1	13210	22909	42972	63973	--
11	10426	26248	--	--	--
2	39716	150988	--	--	--
3	21408	39975	92229	183415	--
10	18360	63584	--	--	--
12	22624	41387	89947	162186	--
Viscosity at 135 C, cSt					
4	553	1219	--	--	--
5	500	--	--	--	--
7	619	--	--	--	--
8	550	793	2707	--	--
1	788	1917	--	--	--
11	770	1210	--	--	--
2	1655	3891	--	--	--
3	1202	1702	2742	3837	--
10	1091	1530	2789	5078	--
12	1140	2132	--	--	--

Table 16. Predicted HP-GPC properties from lab aging test.

Project	PAO	PC1	Y1	Y5	Y10	Y20	Y30	Ult.Prop.
X1, 8-slice, %								
4	13.80	16.69	18.71	19.20	19.3	19.3	19.4	19.4
5	13.30	15.77	--	19.52	--	--	--	--
7	7.39	13.81	17.11	19.29	20.6	21.8	22.3	23.8
8	13.24	17.37	17.02	18.27	19.4	20.8	21.6	25.1
1	11.65	14.24	14.44	16.04	17.3	18.6	19.3	21.6
11	13.25	17.72	17.50	18.71	19.5	20.2	20.5	21.5
2	14.87	17.95	18.22	20.53	--	--	--	--
3	14.32	17.17	18.36	20.15	21.9	24.5	26.3	36.0
10	13.46	16.08	17.75	19.10	20.4	22.1	23.3	28.6
12	14.34	15.55	17.91	19.42	--	--	--	--
X2, 8-slice, %								
4	14.68	15.65	14.74	15.07	15.0	15.0	15.0	15.0
5	14.42	16.30	--	15.04	--	--	--	--
7	12.78	15.40	15.04	15.11	15.1	15.1	15.1	15.1
8	14.83	15.95	15.02	15.04	15.1	15.1	15.1	15.1
1	13.44	15.66	14.32	14.66	14.8	14.9	15.0	15.1
11	14.88	15.09	15.23	15.31	15.4	15.4	15.4	15.5
2	16.42	16.85	17.20	17.61	17.9	18.1	18.3	18.6
3	15.13	15.36	15.38	15.68	15.6	15.6	15.6	15.5
10	15.04	16.00	15.20	15.32	15.3	15.3	15.3	15.3
12	15.29	14.98	15.43	15.54	15.7	15.9	16.1	19.5
X7, 8-slice, %								
4	7.87	6.96	7.27	7.24	7.2	7.2	7.2	7.2
5	8.06	6.36	--	7.18	--	--	--	--
7	10.21	6.95	7.34	7.15	7.1	7.1	7.1	7.1
8	7.85	7.01	7.37	7.29	7.3	7.3	7.3	7.3
1	8.16	7.28	7.52	7.23	7.1	7.1	7.1	7.0
11	7.75	7.17	7.14	7.06	7.1	7.0	7.0	7.0
2	5.92	5.74	5.39	5.09	4.9	4.6	4.5	4.0
3	7.27	6.94	6.76	6.40	5.4	--	--	--
10	7.46	6.98	6.90	6.82	6.8	6.8	6.8	6.8
12	7.27	7.50	6.83	6.69	6.6	6.5	6.5	6.4
X1 + X2, 8-slice, %								
4	28.48	32.34	33.45	34.27	34.3	34.3	34.4	34.4
5	27.72	32.07	--	34.56	--	--	--	--
7	20.17	29.21	32.15	34.40	35.7	36.9	37.5	39.0
8	28.07	33.32	32.04	33.31	34.4	35.8	36.7	40.1
1	25.09	29.90	28.76	30.70	32.1	33.5	34.3	36.7
11	28.13	32.81	32.73	34.02	34.8	35.6	36.0	37.0
2	31.29	34.80	35.42	38.14	--	--	--	--
3	29.45	32.53	33.74	35.83	37.5	40.1	41.8	51.5
10	28.50	32.08	32.95	34.42	35.7	37.5	38.6	43.9
12	29.63	30.53	33.34	34.96	--	--	--	--

Table 17. Regression analyses between TMA and HP-GPC parameters (n=73).

Dependent Variables	LMS+MMS1		LMS		3-SLICE	
	P-value	R**2	P-value	R**2	P-value	R**2
Tg	0.3710	0.011	0.1655	0.027	0.7365	0.018
Tsp	0.0022	0.124	0.1233	0.033	0.0160	0.138
ML	0.0475	0.054	0.1597	0.028	0.1046	0.085
MH	0.6908	0.002	0.8232	0.001	0.2022	0.064

Dependent Variables	4-SLICE		8-SLICE		MWT+POLYIDX	
	P-value	R**2	P-value	R**2	P-value	R**2
Tg	0.3455	0.063	0.0381	0.216	0.3836	0.027
Tsp	0.0081	0.181	0.0033	0.292	0.0004	0.202
ML	0.0517	0.127	0.0037	0.289	0.1665	0.050
MH	0.3904	0.058	0.3786	0.120	0.4485	0.023

Dependent Variables	Selected variables by stepwise regressions
Tg	None
Tsp	LMS+MMS1, MMS1, X2, MWT, POLYIDX
ML	MMS2, LMS, MMS2, X5, MWT
MH	None



Table 18. Regression analyses between Physical properties and TMA parameters (n=80).

Dependent Variables	Tg		Tsp		ML		MH		ALL 4 PARAMETERS		Selected variables from stepwise reg.
	P-value	R**2	P-value	R**2	P-value	R**2	P-value	R**2	P-value	R**2	
Rheological properties											
P5	0.0003	0.152	0.0001	0.228	0.0106	0.081	0.6021	0.004	0.0001	0.357	ALL
P25	0.0125	0.077	0.0001	0.207	0.0027	0.110	0.9885	0.000	0.0001	0.292	Tsp
P4	0.0016	0.121	0.0001	0.246	0.0057	0.094	0.8209	0.001	0.0001	0.340	ALL
VIS25	0.0037	0.103	0.0001	0.399	0.0003	0.157	0.7735	0.001	0.0001	0.474	Tsp
CF	0.9515	0.000	0.0001	0.249	0.0001	0.173	0.7922	0.001	0.0001	0.487	ALL
SI	0.9571	0.000	0.0001	0.249	0.0013	0.125	0.5032	0.006	0.0001	0.465	ALL
VIS60	0.2055	0.020	0.0001	0.297	0.0009	0.133	0.6978	0.002	0.0001	0.430	Tsp, ML, MH
VIS135	0.0520	0.048	0.0001	0.358	0.0001	0.173	0.7772	0.001	0.0001	0.508	Tsp, ML, MH
SP	0.0372	0.054	0.0001	0.336	0.0003	0.158	0.8249	0.001	0.0001	0.426	Tsp, ML, MH
Temperature susceptibility											
PR	0.5794	0.004	0.0004	0.150	0.0008	0.135	0.4413	0.008	0.0002	0.249	ALL
PI	0.1248	0.030	0.1208	0.031	0.3939	0.009	0.6061	0.003	0.0569	0.114	Tg, Tsp
CN	0.8330	0.001	0.0376	0.054	0.0960	0.035	0.6208	0.003	0.0497	0.118	Tsp
VTS	0.9091	0.000	0.5999	0.004	0.6046	0.003	0.8512	0.000	0.9374	0.011	None
PVN60	0.9802	0.000	0.0107	0.081	0.0413	0.052	0.7488	0.001	0.0083	0.165	Tsp
PVN135	0.9441	0.000	0.0970	0.035	0.2008	0.021	0.8359	0.001	0.2380	0.070	Tsp
Low-temperature cracking properties											
CT	0.0036	0.104	0.2035	0.021	0.9654	0.000	0.2546	0.017	0.0125	0.155	Tg, MH
TES	0.0016	0.120	0.0022	0.114	0.0216	0.066	0.8811	0.000	0.0008	0.221	Tg, Tsp
S23	0.0002	0.161	0.0001	0.280	0.0006	0.140	0.9183	0.000	0.0001	0.402	ALL
S29	0.0009	0.133	0.0001	0.211	0.0009	0.133	0.9954	0.000	0.0001	0.345	ALL

Table 19. Regression analyses between Physical properties and HP-GPC parameters (n=73).

Dependent Variables	LMS		LMS+MMS1		3-SLICE		4-SLICE		8-SLICE		Selected variables from stepwise reg.
	P-value	R**2	P-value	R**2	P-value	R**2	P-value	R**2	P-value	R**2	
Rheological properties											
P5	0.0004	0.161	0.0001	0.245	0.0001	0.333	0.0001	0.356	0.0001	0.568	X2,X4,X5,X7,X8
P25	0.0001	0.248	0.0001	0.385	0.0001	0.432	0.0001	0.429	0.0001	0.549	X4,X6,X7,X8
P4	0.0001	0.191	0.0001	0.298	0.0001	0.378	0.0001	0.389	0.0001	0.560	X2,X7
VIS25	0.0012	0.139	0.0001	0.253	0.0001	0.302	0.0002	0.278	0.0001	0.462	X4,X6,X7,X8
CF	0.0500	0.053	0.0001	0.283	0.0001	0.339	0.0001	0.475	0.0001	0.546	X2
SI	0.0173	0.077	0.0001	0.311	0.0001	0.369	0.0001	0.457	0.0001	0.509	X2,X4
VIS60	0.0421	0.057	0.0001	0.185	0.0006	0.221	0.0006	0.249	0.0001	0.325	X2
VIS135	0.0036	0.113	0.0001	0.361	0.0001	0.416	0.0001	0.475	0.0001	0.588	X7,X8
SP	0.0001	0.208	0.0001	0.353	0.0001	0.391	0.0001	0.367	0.0001	0.505	X4,X6,X7,X8
Temperature susceptibility											
PR	0.0004	0.161	0.0001	0.258	0.0001	0.278	0.0001	0.307	0.0001	0.420	X5
PI	0.2533	0.018	0.1021	0.037	0.0523	0.105	0.0282	0.146	0.0366	0.218	X3,X4,X6
CN	0.1110	0.035	0.0208	0.073	0.1480	0.074	0.1827	0.086	0.1447	0.166	X5
VTS	0.1798	0.025	0.6095	0.004	0.8754	0.010	0.4803	0.049	0.2465	0.142	X3
PVN60	0.2874	0.016	0.0099	0.090	0.0700	0.097	0.0061	0.188	0.0076	0.268	X2,X8
PVN135	0.7255	0.002	0.0619	0.048	0.2190	0.062	0.0001	0.301	0.0001	0.433	X2,X8
Low-temperature cracking properties											
CT	0.2700	0.017	0.2242	0.021	0.0515	0.106	0.0103	0.174	0.0001	0.311	X1,X2,X7
TES	0.0001	0.205	0.0001	0.273	0.0001	0.317	0.0001	0.354	0.0001	0.432	X2,X7
S23	0.0032	0.116	0.0001	0.236	0.0001	0.334	0.0001	0.326	0.0001	0.411	X2,X7
S29	0.0099	0.090	0.0001	0.211	0.0001	0.319	0.0001	0.316	0.0001	0.467	X1,X2,X7,X8

Table 20. Regression analyses:  
physical properties against TMA and HP-GPC parameters (n=73).

Dependent Variables	TMA & HP-GPC parameters		Selected variables from stepwise reg.	
	P-value	R**2	TMA parameters	HP-GPC parameters
Rheological properties				
P5	0.0001	0.666	<b>Tsp</b>	<b>X2,X6,X7</b>
P25	0.0001	0.669	Tsp,ML,MH	<b>X4,X6,X7</b>
P4	0.0001	0.667	Tsp	<b>X2,X4,X6,X7,X8</b>
VIS25	0.0001	0.766	<b>Tsp</b>	<b>X2,X4,X6,X7,MWT,PIDX</b>
CF	0.0001	0.741	Tg,Tsp, <b>ML,MH</b>	<b>X2</b>
SI	0.0001	0.719	Tg, <b>Tsp</b> ,ML,MH	<b>X2</b>
VIS60	0.0001	0.583	<b>Tsp</b>	<b>X8</b>
VIS135	0.0001	0.773	<b>Tsp</b> ,ML,MH	<b>X7</b>
SP	0.0001	0.715	<b>Tsp</b> ,ML,MH	<b>X1,X3,X6,MWT</b>
Temperature susceptibility				
PR	0.0001	0.636	Tsp	<b>X2,X4,X5</b>
PI	0.0517	0.309	Tg,Tsp	<b>X4,PIDX</b>
CN	0.2076	0.246	Tsp	<b>X5</b>
VTs	0.5121	0.187		<b>X3</b>
PVN60	0.0098	0.368		<b>X2,X8</b>
PVN135	0.0001	0.496		<b>X2,X8</b>
Low-temperature cracking properties				
CT	0.0008	0.438	Tg	<b>X2,X5,X7,X8</b>
TES	0.0001	0.564	Tg	<b>X2,X7</b>
S23	0.0001	0.655	<b>Tsp</b> ,ML,MH	<b>X2,X4,X7,PIDX</b>
S29	0.0001	0.588	Tsp,ML,MH	<b>X2,X5,X7</b>

Bold face indicates significantly correlated variable.

Table 21. Durability Tests on Neat Asphalts

Year	Name	Test Method	Field Correlation	Ref
1897	Dow	Heating 20 g of asphalt in a 2-oz glass retort at 400 F for 30 hrs.	No	14, 66
1905	Loss on heating	A 50 g sample is placed in a 3-oz can and maintained at 325 F for 5 hrs in a standard revolving shelf oven	No	51
1937	Nicholson	Air-blown at 425 F for 15 min. using air at 1/3 cu ft per min. Or air blow the asphalt to a pen of 20-25.	No	51
1937	Raschig and Doyle	Air-blown at 400 F for 15 min. using air at 1/3 cu ft per min.	No	51
1937	Hubbard & Gollomb	Varied time and temperature	No	51
1937	Benson	Translucent asphalt film 0.001 in. thick exposed to natural and artificial light and heat and observed under microscope and classified.	No	51
1941	Lewis & Welborn (TFOT)	A 50 ml sample is heated in a 1/8 in. film in a 5.5 in. flat container for 5 hr at 325 F.	Changes in asphalt equivalent to hot-plant mixing	51
1942	Anderson, Stross & Ellings	Resistance to hardening is defined based on penetrations of residue of loss-on-heating tests after 5 and 10 hr heating at 325 F	No asphalts with poor service records were found with a resistance to hardening value below 55	2
1942	Anderson, Stross & Ellings	Asphalt dissolved in benzene is oxidized in an oxygen bomb at 108 psi and at 50 C for 40 hrs. At the end of the run the asphalt is recovered and penetration determined. A "deterioration index" is calculated based on penetration and pressure drops.	An index lower than 15 indicated satisfactory performance; poor durability was associated with an index above 20.	2
1955	Griffin, Miles & Penther (Shell Microfilm Aging Test)	A 5-micron film of asphalt was aged on glass plates for 2 hrs at 225 F.	Predicts relative rates of field hardening based on aging index (viscosity before and after aging).	28, 29, 51, 63

1959	Blokker and van Hoorn	Treating bitumen in thin film (5 to 200-micron) in oxygen bomb of 20 atm (300 psi) at 50 C.	One day aging in the bomb is equivalent to 6 months on the road	6
1961	Traxler	Modified Shell microfilm test: a 15-micron film at 225 F for 2 hrs	Equivalent to TFOT	65
1963	Hveem, Zube and Skog	Modified Shell microfilm test: a 20-micron film at 210 F for 24 hrs.	Yes	31
1963	Hveem, Zube and Skog (RTFOT)	A 35 g sample is placed in a bottle and exposed to controlled amount of air circulation in an oven at 325 F for 85 min.	Equivalent to TFOT	31
1966	Davis and Petersen	Column oxidation: 15-micron asphalt film coated on Teflon particles and aged in a gas chromatographic column at 130 C for 24 hrs. by passing air through the column.	Limited	13
1967	Skog	Expose asphalts first to RTF exposure (325 F, 75 min.) followed by microfilm aging (20-micron, 210 F, 2 hrs.)	5 yr. field service hardening under California weathering conditions.	64
1969	Lee, Iowa Durability Test (IDT)	Residue from the TFOT was exposed to a pressure-oxidation treatment of 20 atm oxygen at 150 F for up to 1000 hrs.	46 hrs in the IDT aged asphalts to the equivalent of 60 months of service in Iowa.	47
1969	Schmidt and Santucci, Rolling thin film circulating (RTF-C) oven test	Modified RTFOT: 20-micron film, 210 F for 48 hrs in a forced-draft (circulating) oven. Asphalt film deposited in the bottle via asphalt-in-benzene solution.	Zaca-Wigmore test road	40, 51, 62
1981	Kemp and Predoehl, (California Tilt-Oven Durability Test)	Modified RTFOT: 35 g, 113 C in a RTF oven tilted 1.06 degrees for 168 hrs. (7 days)	2-year Aging in hot desert sites in California	41
1985	Edler et al	Extended RTFOT (8 hrs) followed by further aging of 30-micron asphalt films in a POB at 300 psi oxygen and 65 C for 96 hrs.	No	16

1987	Kim et al	Oxygen bomb: POB at 100 psi and 60 C on Fraass brittle test samples, 0.02 in thick, for 1 to 5 days.	Limited: 5-days in POB gave Fraass brittle points and compositions equivalent to 5-10 years in Oregon.	43
1989	FHWA	Forced air distillation (FAD)	Changes in asphalts during drum-dryer and batch mixing operations	12
1989	FHWA	Revolving forced air distillation (RFAD)	Changes in asphalts during drum-dryer and batch mixing operations	12
1989	FHWA	Small steam distillation (SSD): modified ASTM D255	Changes in asphalts during drum-dryer and batch mixing operations	12
1989	Petersen	Thin film accelerated aging test (TFAAT): A modification of RTF-C. A 4.0 g. (160-micron) of asphalt is deposited into RTFOT bottle by 10 ml toluene, heated to 113 C (235 F) for 3 days.	Similar to asphalt aging in 11-13 years old pavements at 6-8% air.	56

Table 22. Durability Tests on Mixtures

Year	Name	System	Test Method	Field Correlation	Ref
1897	Dow	sand asphalt	Mix aged in oven at 300 F for 30 min.	No	14
1937	Hubbard & Gollomb	sand asphalt	Varied time and temperature	No	51
1940	Shattuck mixing test	sand asphalt	A 2 kg sample of 94% Ottawa sand and 6% asphalt mixed in the lab for one min. at 275-300 F. Mixture heated at 350 F for 30 min. in a pan 7x11 in.	Changes in asphalt equivalent to plant mixing	51
1952	Pauls and Welborn	sand asphalt	Effect of time on compressive strength of 2x2 in cylinder specimens of Ottawa sans asphalt mixes heated at 325 F	No	55
1963	Hveem et al.	sand asphalt	Subjecting 2% asphalt Ottawa sand mixtures to infrared radiation in a weathering oven at 140 F.	1000 hrs in weathering machine produced hardening equivalent to 5 yrs of service life.	31
1965	Goode and Lufsey	Marshall samples	Aged in oven at 140 F for 12 days	8-years in-service aging	24, 54
1987	Kim et al	Compact-ed mixes	Mixes compacted to 6 and 12% air by kneading compactor, aged in pressure-oxidation bomb (POB) at 100 psi oxygen and 60 C for up to 5 days	5 days in POB equivalent to field aging of 5 to 10 years.	43

Table 23. Critical values for performance related parameters.

Property	Critical Value	Reference	Corresponding		Corresponding		
			X2,%	X7,%	Tg,C	Tsp,C	ML,um/C
P4	>5	Goodrich, 1988	<18.5	> 4.5	< -9.5	<32.9	<0.51
P25	>20	Finn, 1967	<18.0	> 5.0	<-18.5	<31.0	<0.39
SI	<0.55	Kandhal et al., 1973	<18.1	> 4.6		<36.0	<0.60
R&B SP	<65.5 C	Finn, 1967	<18.7	> 4.6		<31.2	<0.45
VIS25	<20 mega poise	Finn, 1967	<18.2	> 5.2	<-17.5	<28.0	<0.42
PVN60	>-1.3	Goodrich & dimple, 1986	> 9.8	<13.6			
PVN135	>-1.0	McLeod, 1989	>11.1	<12.7			
Stiffness at -23 C and 10,000 sec	<20 ksi	Kandhal, 1978	<19.5	> 3.7	<-17.0	<32.5	<0.47



Table 24. Proposed Trial Specification for Asphalt Cement

Test	AC-5	AC-10	AC-20
<b>Original Asphalt:</b>			
Viscosity @ 60 C, poises*	500+/-100	1000+/-200	2000+/-400
Viscosity @ 135 C, cSt, min.*	175	250	300
Penetration, 25 C, min.*	140	80	60
Flash point, C, min.*	177	219	232
Solubility in trichlo, % min.*	99.0	99.0	99.0
<b>Residue from TFOT:</b>			
Viscosity, 60 C, p., max.*	2000	4000	8,000
<b>Residue from pressure-oxidation, 46 hrs. @ 150 F:</b>			
Viscosity at 60 C, poises, max.	10,000	20,000	40,000
Penetration, 25/100/5, min.	20	20	20
Penetration, 4/200/60, min.	5	5	5
Penetration, 5/100/5, min.	10	8	7
Softening point, F, max.	160	160	160
Stiffness, -23 C, 10,000 sec., psi	20,000	20,000	20,000
Viscosity, 25 C, megapoises, max.	20	20	20
Shear susceptibility, max.	0.55	0.55	0.55
X2 (HP-GPC), %, max.	20	20	20
X7 (HP-GPC), %, min.	5	5	5
Tg(TMA), C, max.	-10	-10	-10
Tsp(TMA), C, max.	28	28	28
ML(TMA), max.	0.4	0.4	0.4

\*AASHTO M226, Table 2

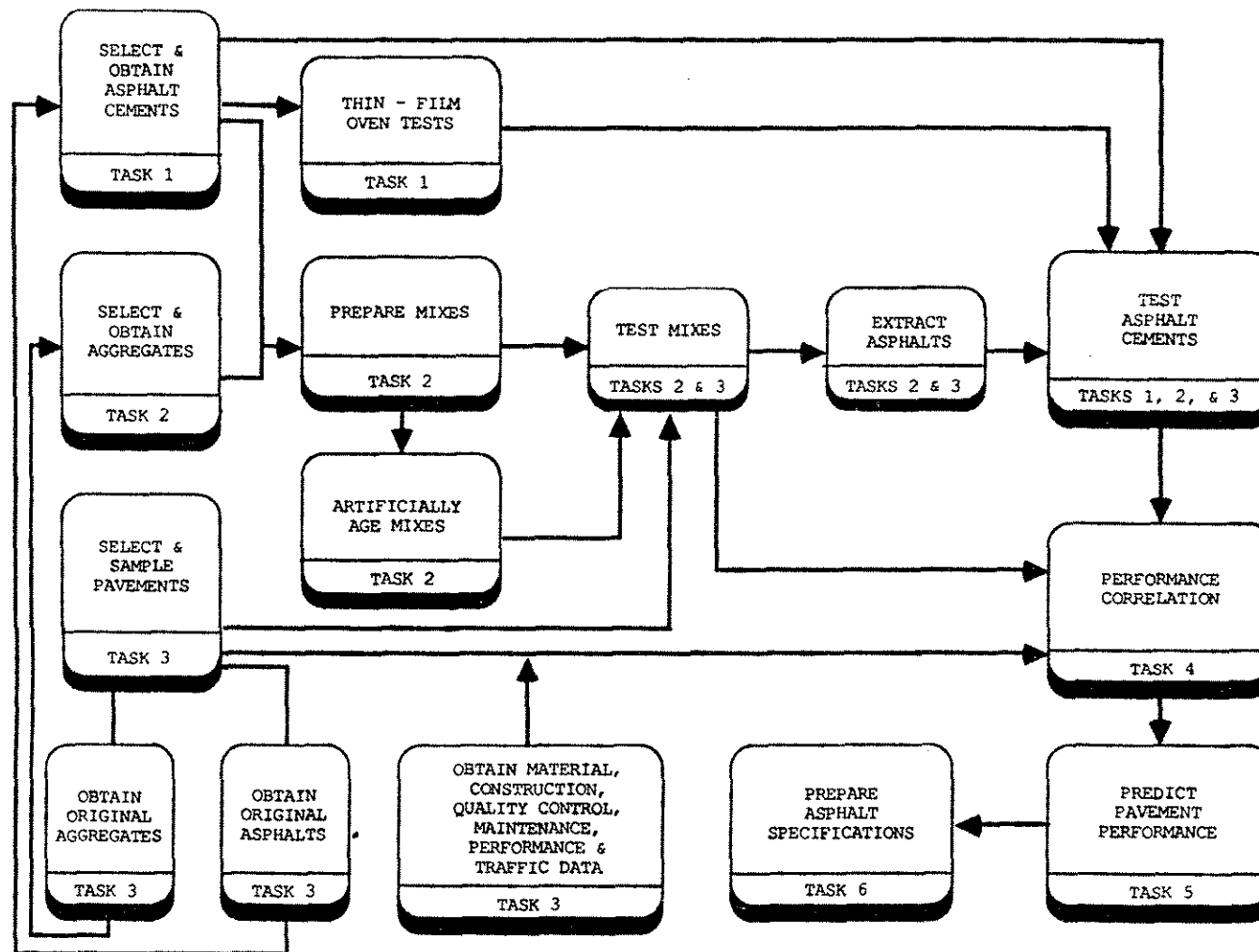


FIGURE 1. SUMMARY OF PROPOSED RESEARCH

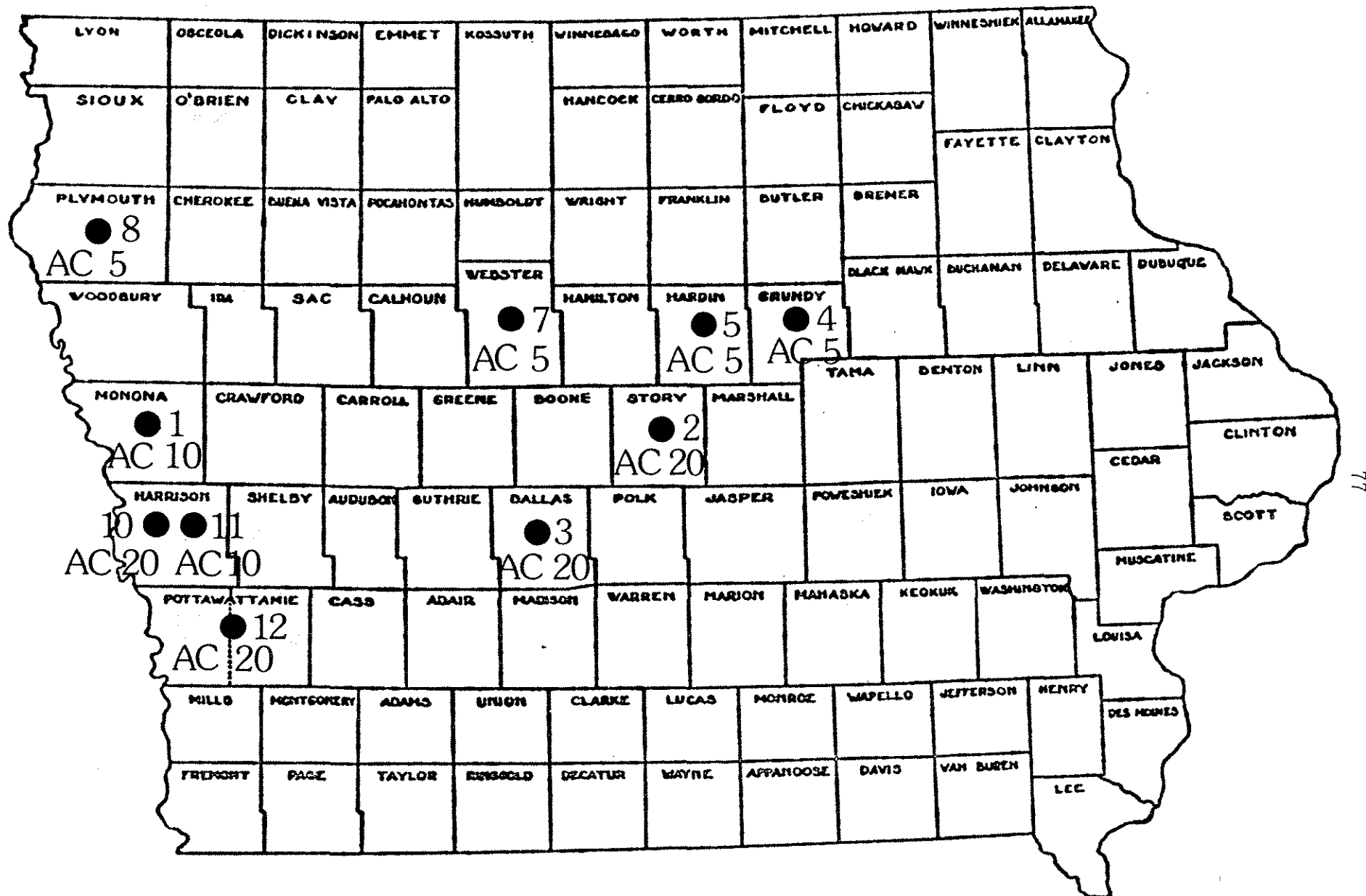
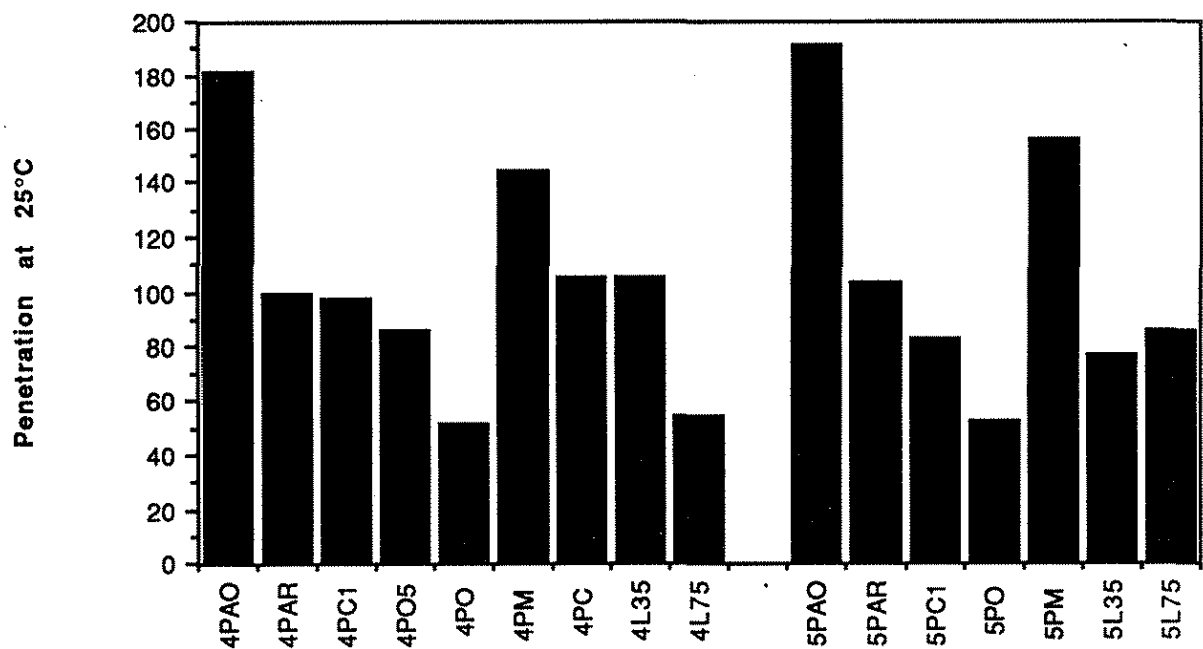


Figure 2. Pavement project locations.

## PROJECT 4 &amp; 5, AC-5



## PROJECT 7 &amp; 8, AC-5

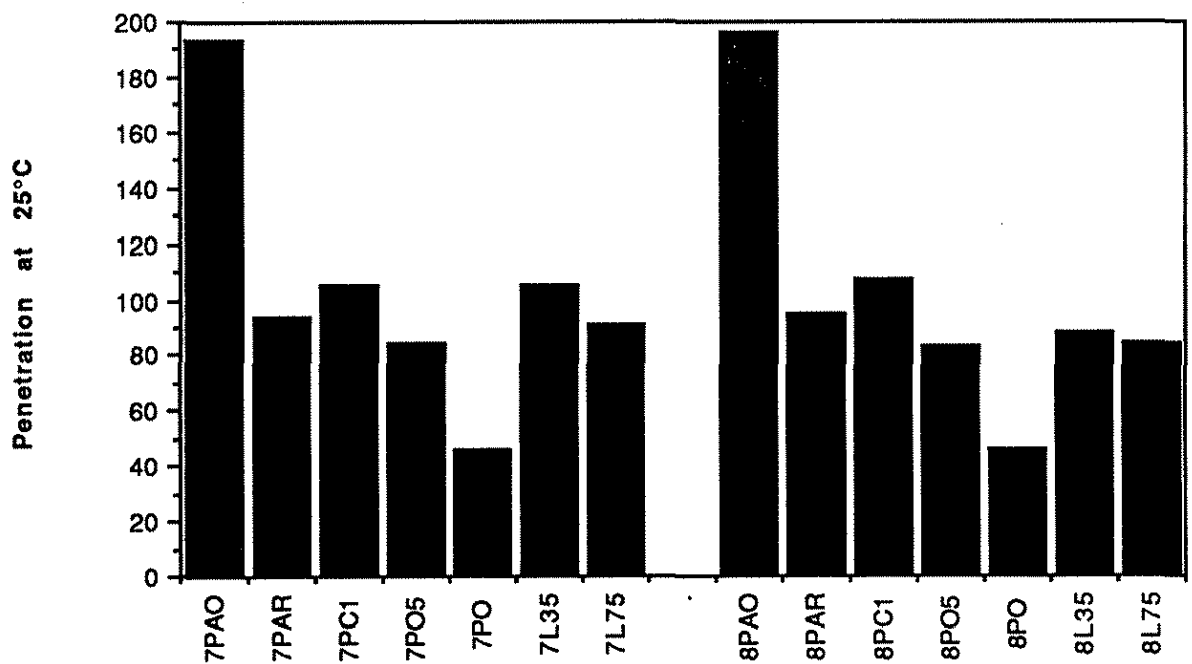


Figure 3. Penetration at 25°C.

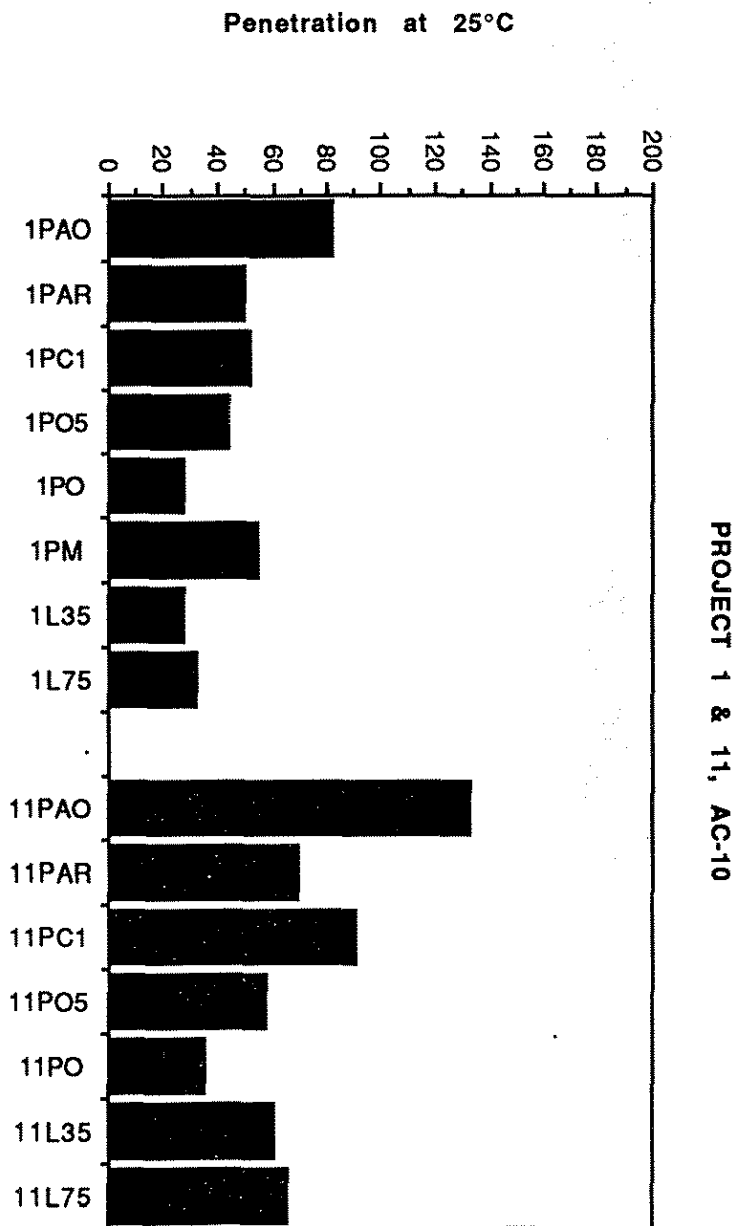
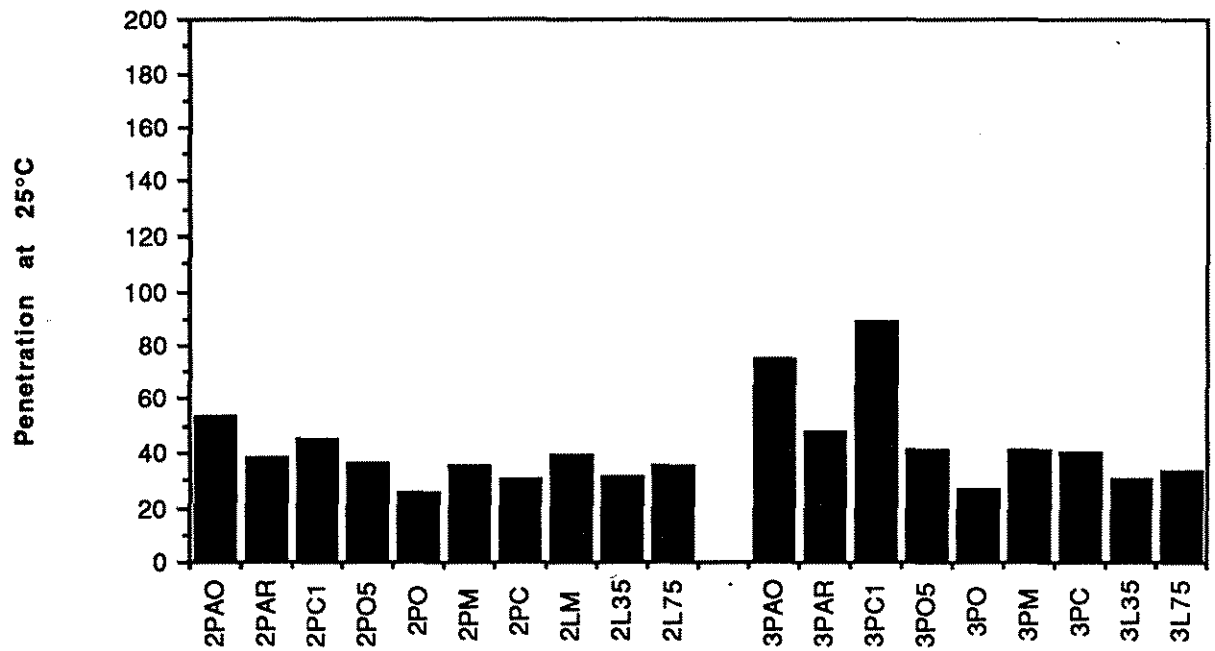


Figure 3. Penetration at 25°C (continued).

## PROJECT 2 &amp; 3, AC-20



## PROJECT 10 &amp; 12, AC-20

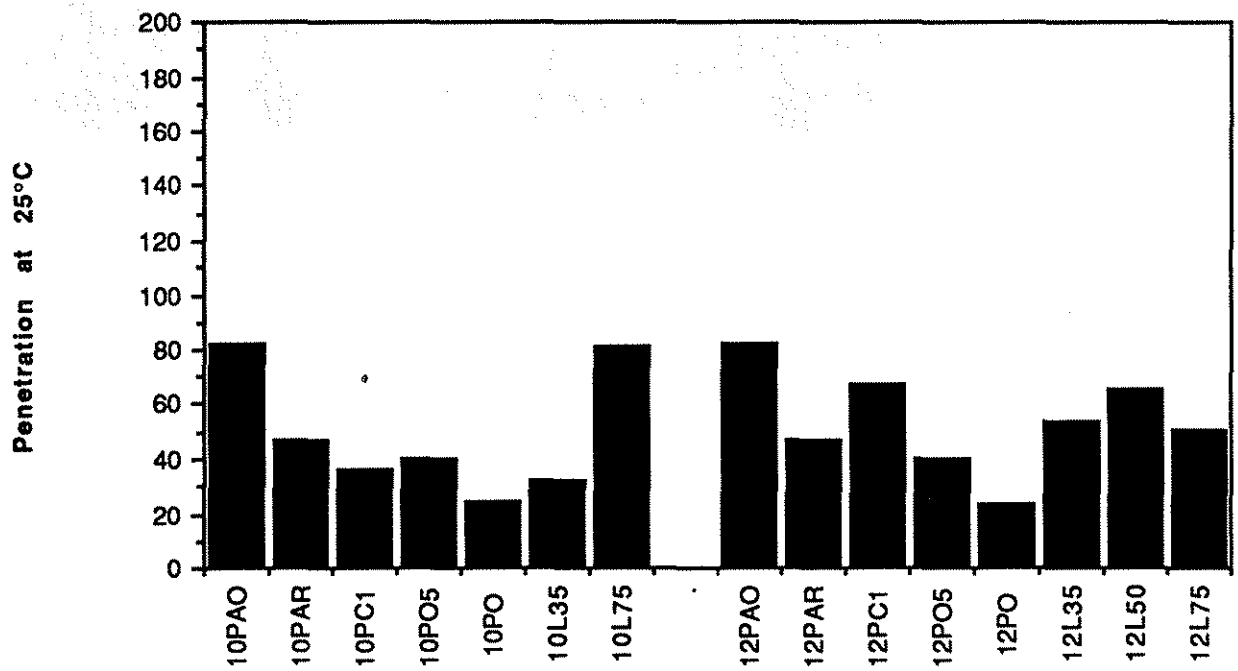
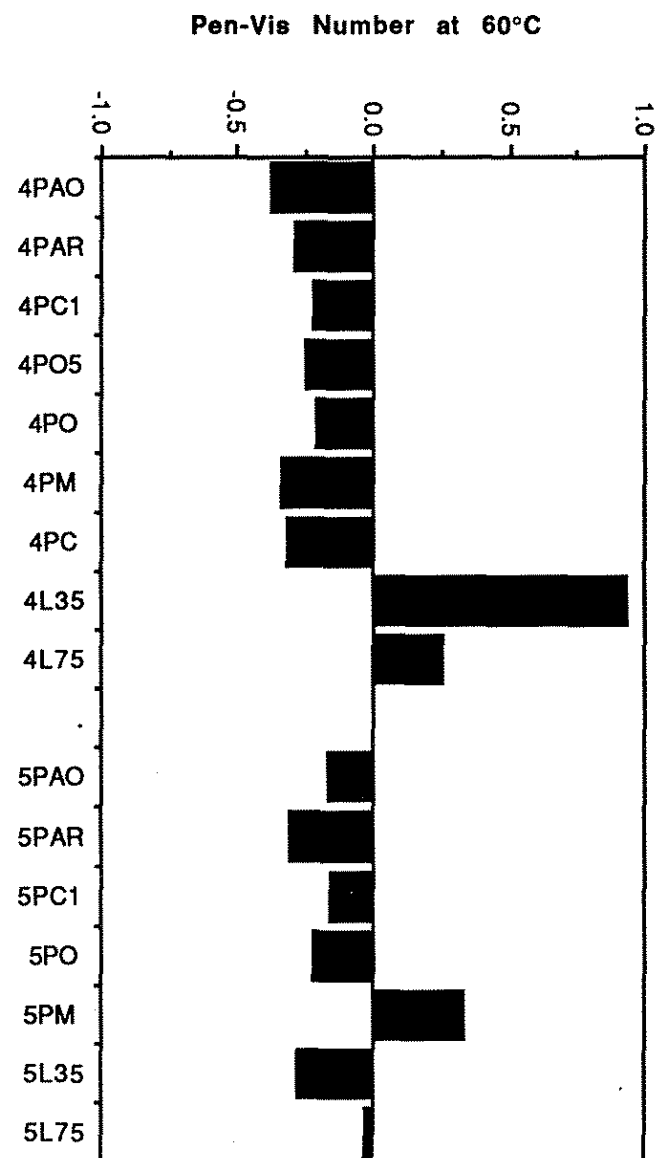


Figure 3. Penetration at 25°C (continued).

## PROJECT 4 &amp; 5, AC-5



## PROJECT 7 &amp; 8, AC-5

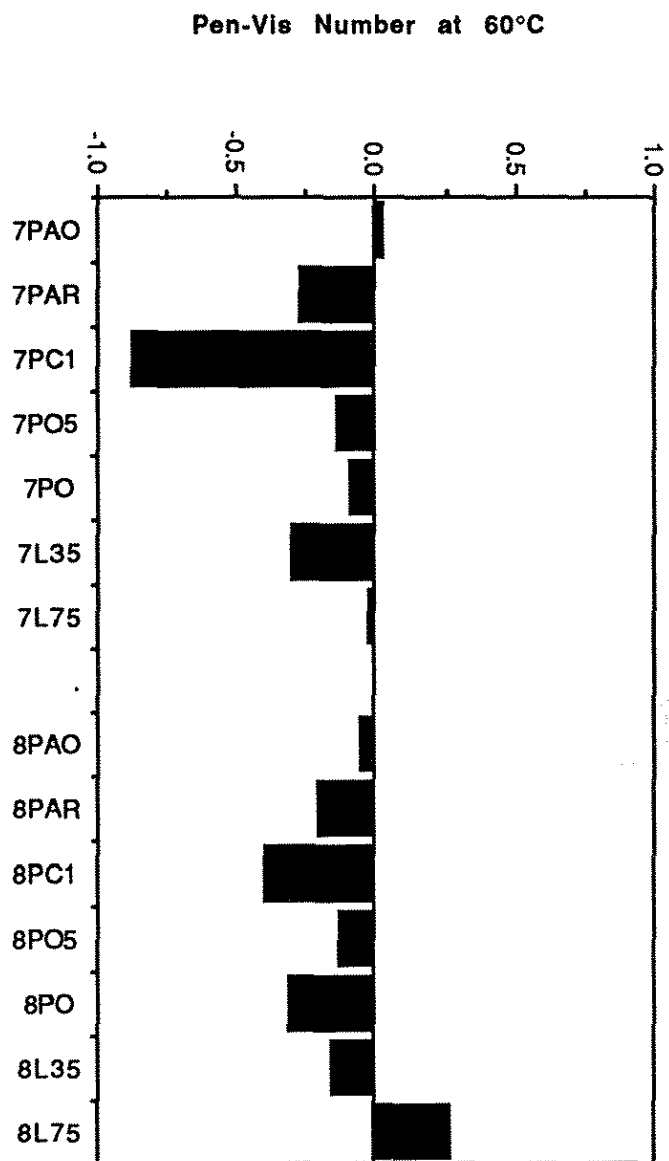


Figure 4. PVN at 60°C.

## PROJECT 1 &amp; 11, AC-10

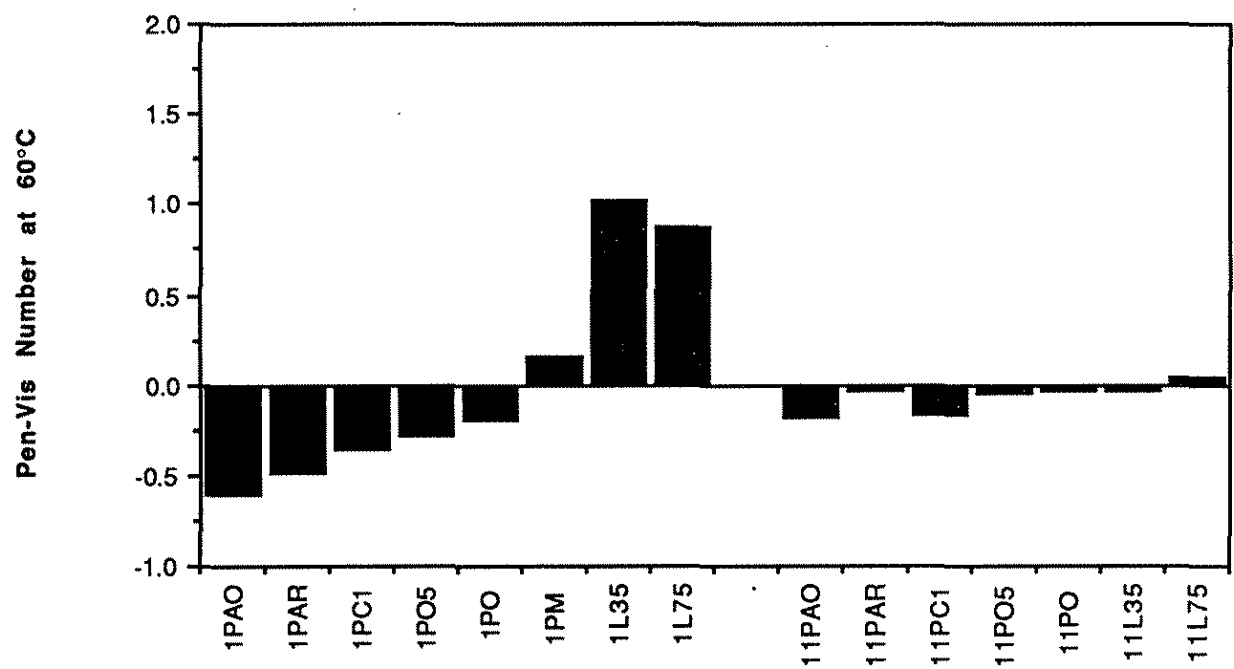
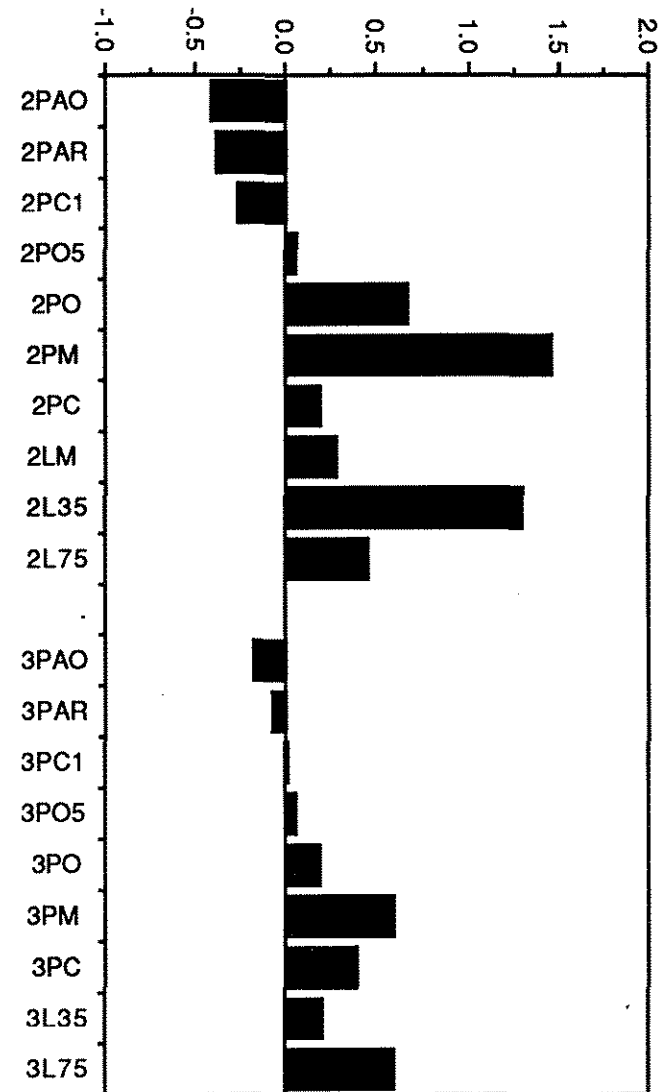


Figure 4. PVN at 60°C (continued).

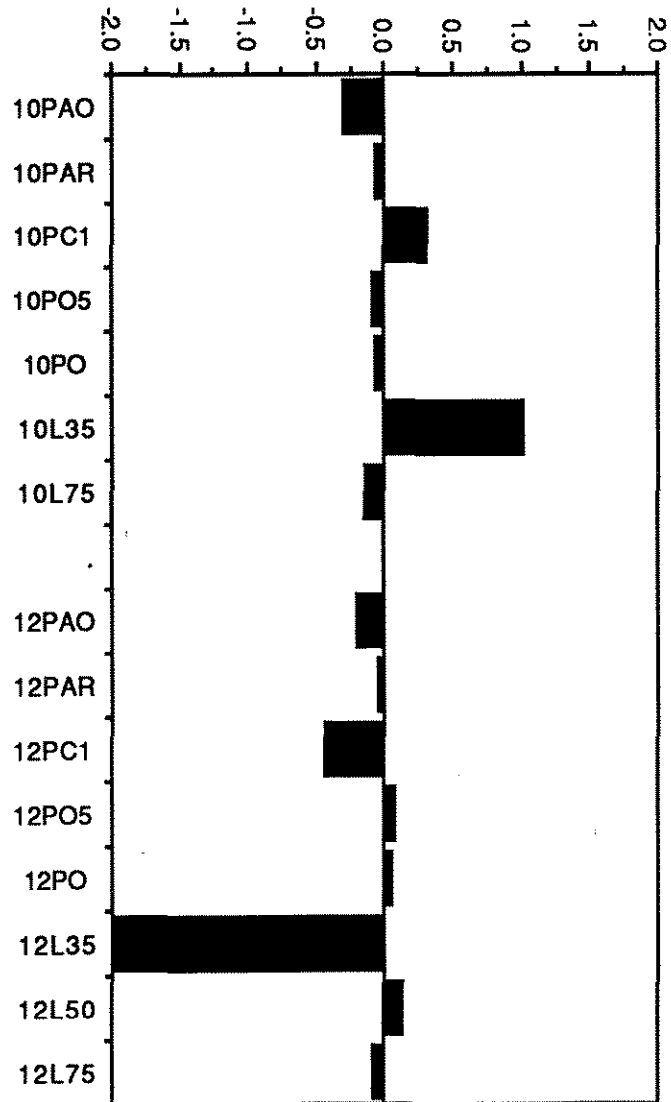


Pen-Vis Number at 60°C



PROJECT 2 &amp; 3, AC-20

Pen-Vis Number at 60°C



PROJECT 10 &amp; 12, AC-20

Figure 4. PVN at 60°C (continued).

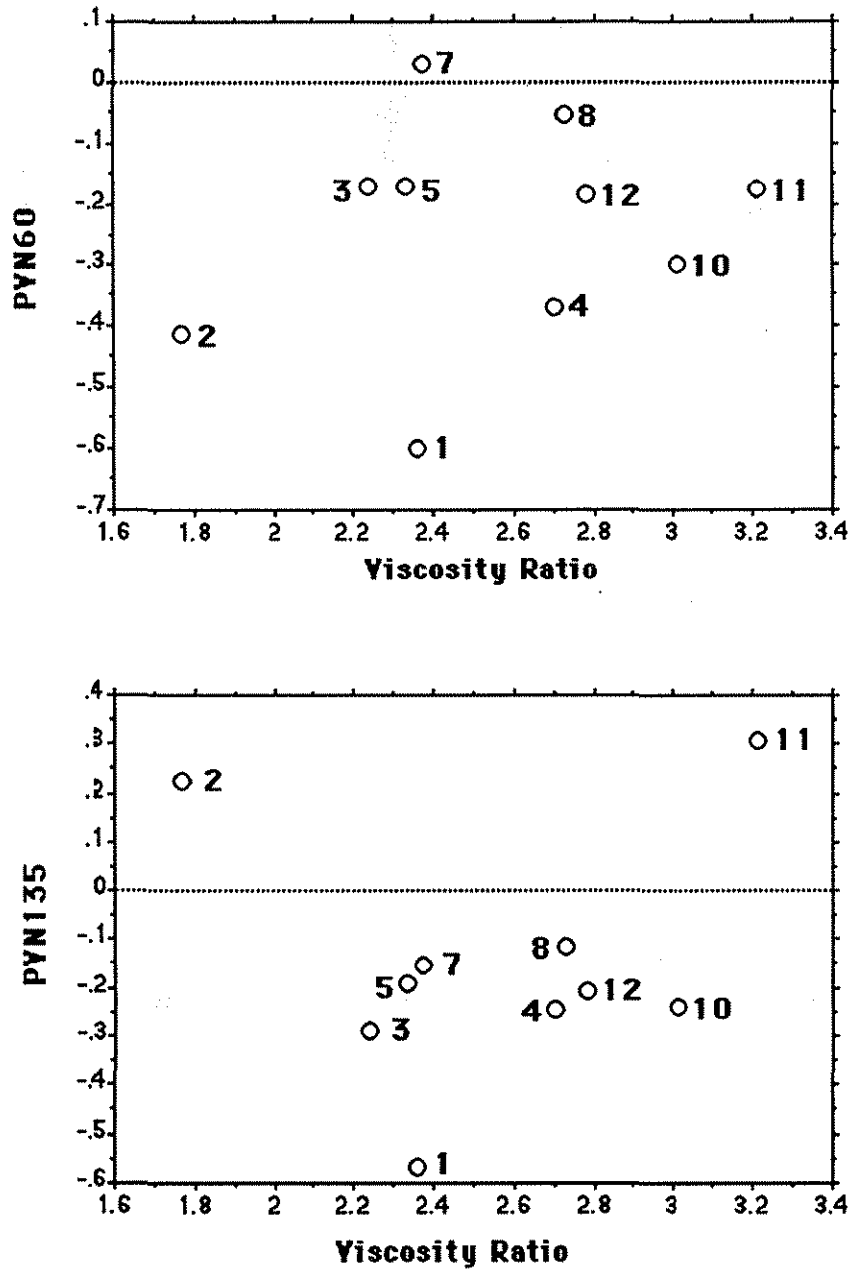


Figure 5. PVN vs viscosity ratio at 60°C.

Sampler: K20-0-01  
Size: 3.00 MM  
Rate: 5 DEG/MIN  
Program: TMA Analysis V2.0

TMA

Date: 2-Feb-89 Time: 13:28:37  
File: DATA.26 TMA 5  
Operator: STEVE  
Plotted: 8-Jan-90 4:24:04

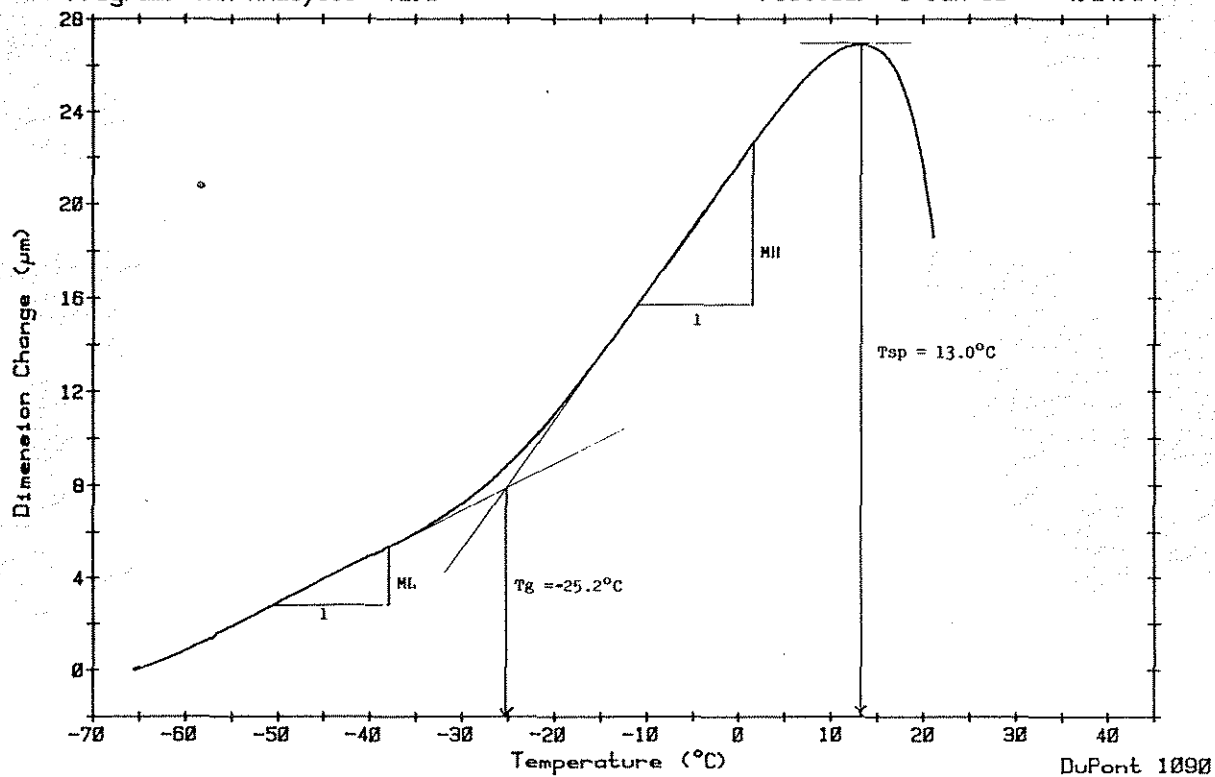


Figure 6. Typical TMA thermogram (K20-01-0).

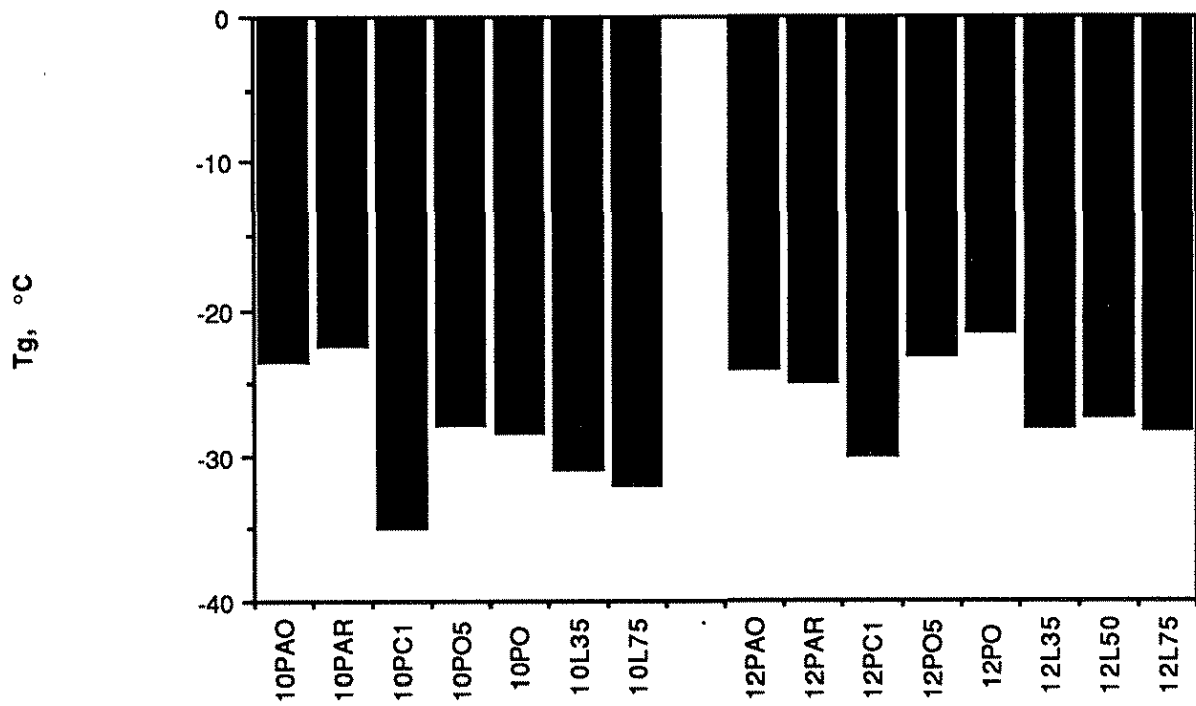


Figure 7. Glass transition temperatures (Tg), asphalts 10 and 12.

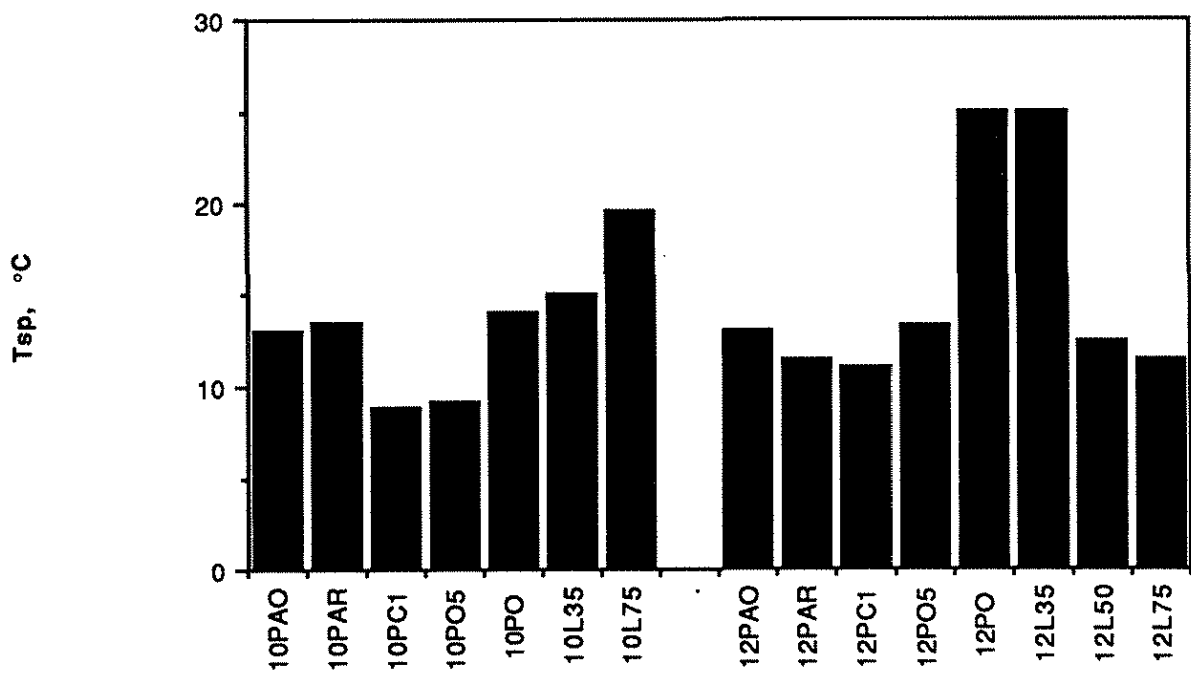


Figure 8. Softening temperatures (Tsp), asphalts 10 and 12.

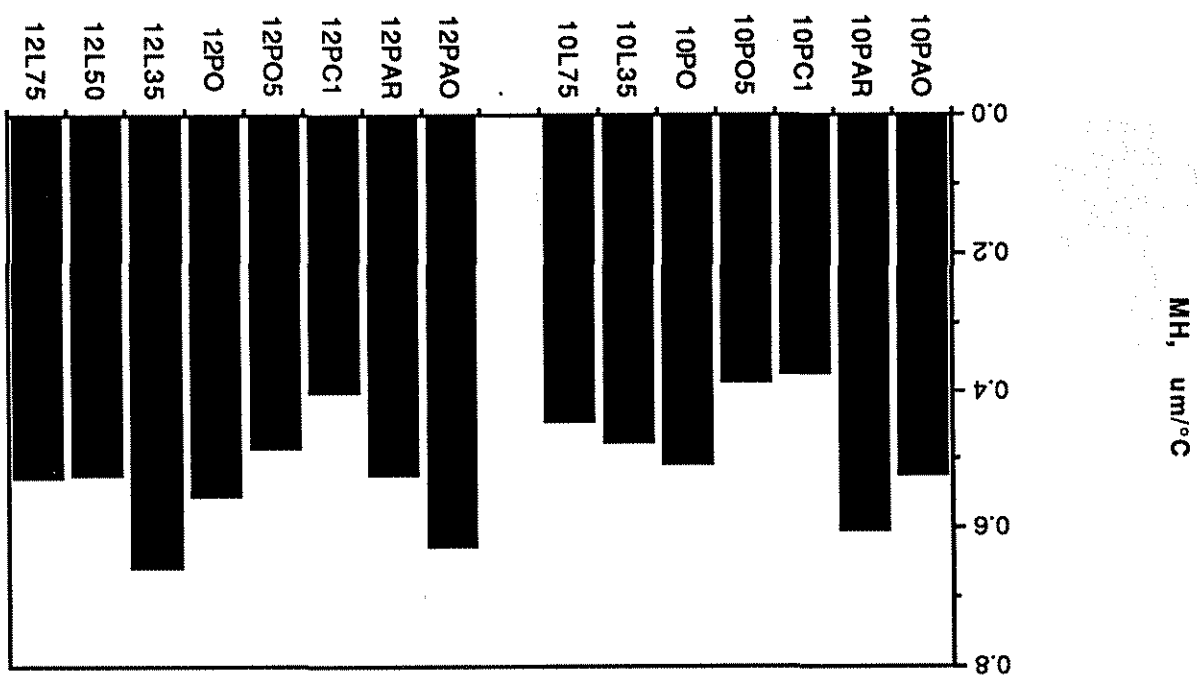


Figure 10. Intermediate temperature expansion slopes (MH), asphalts 10 and 12.

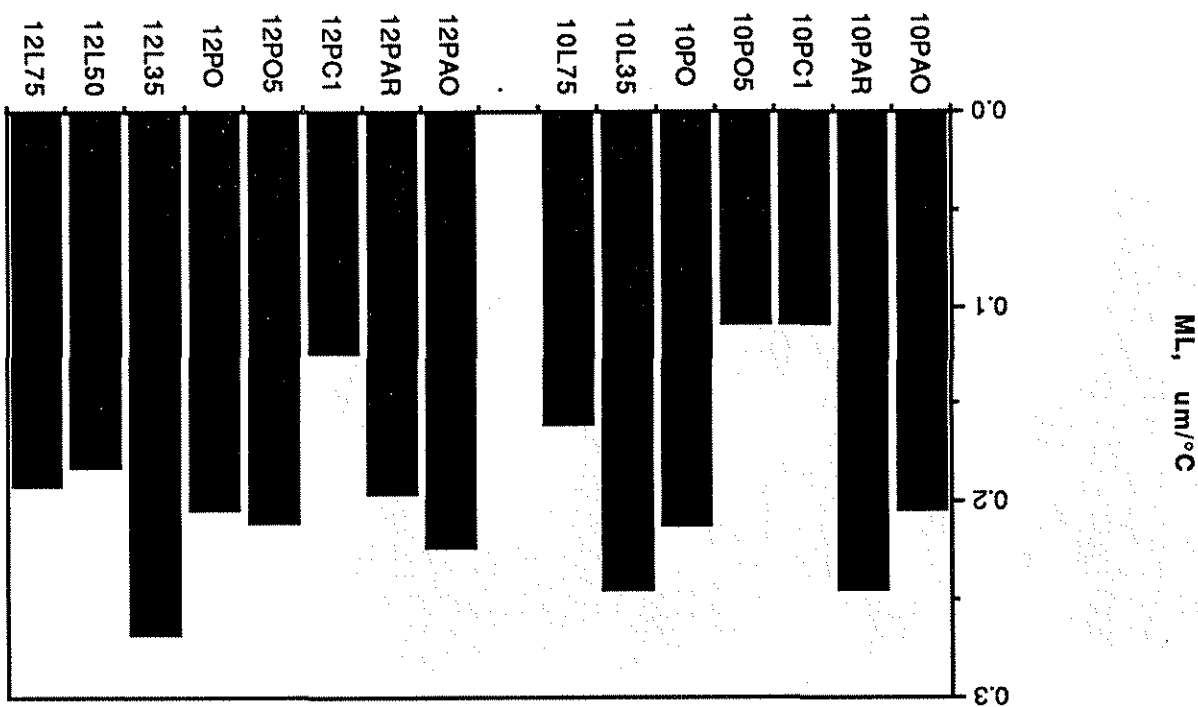


Figure 9. Low-temperature expansion slopes (ML), asphalts 10 and 12.

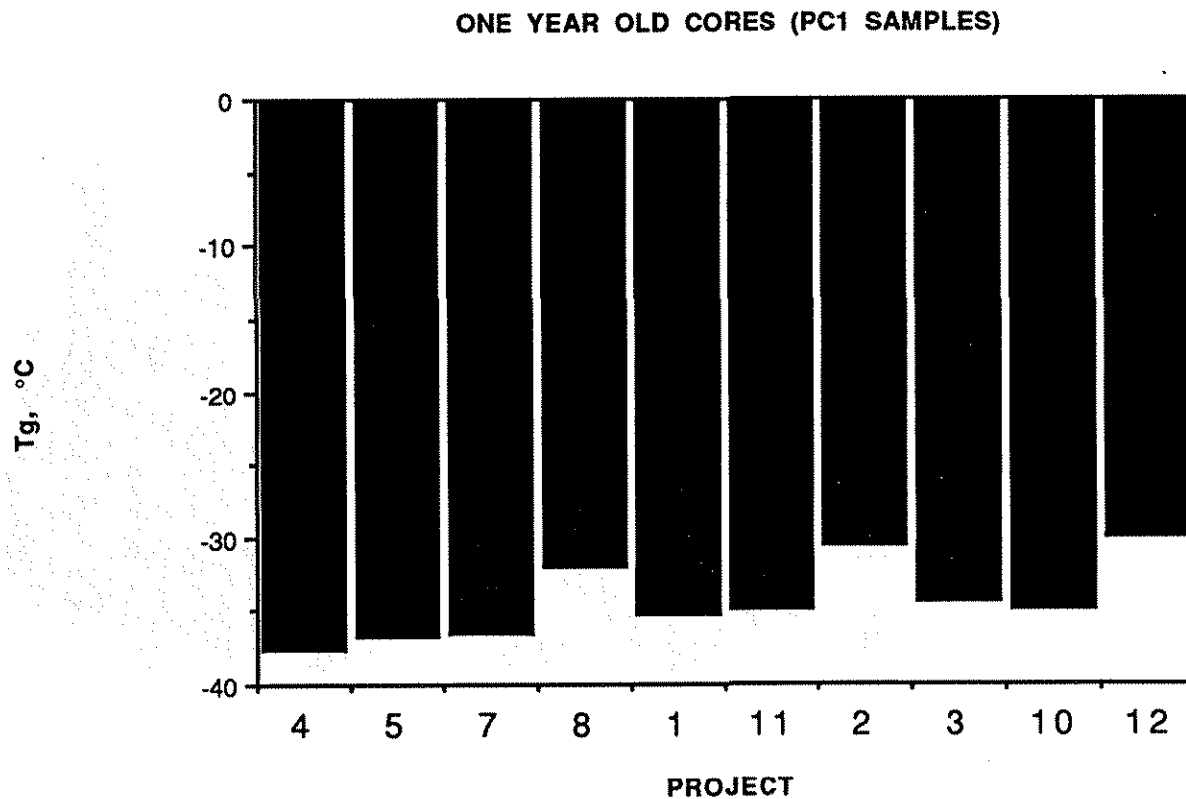


Figure 11. Glass transition temperatures (T<sub>g</sub>), one-year old cores.

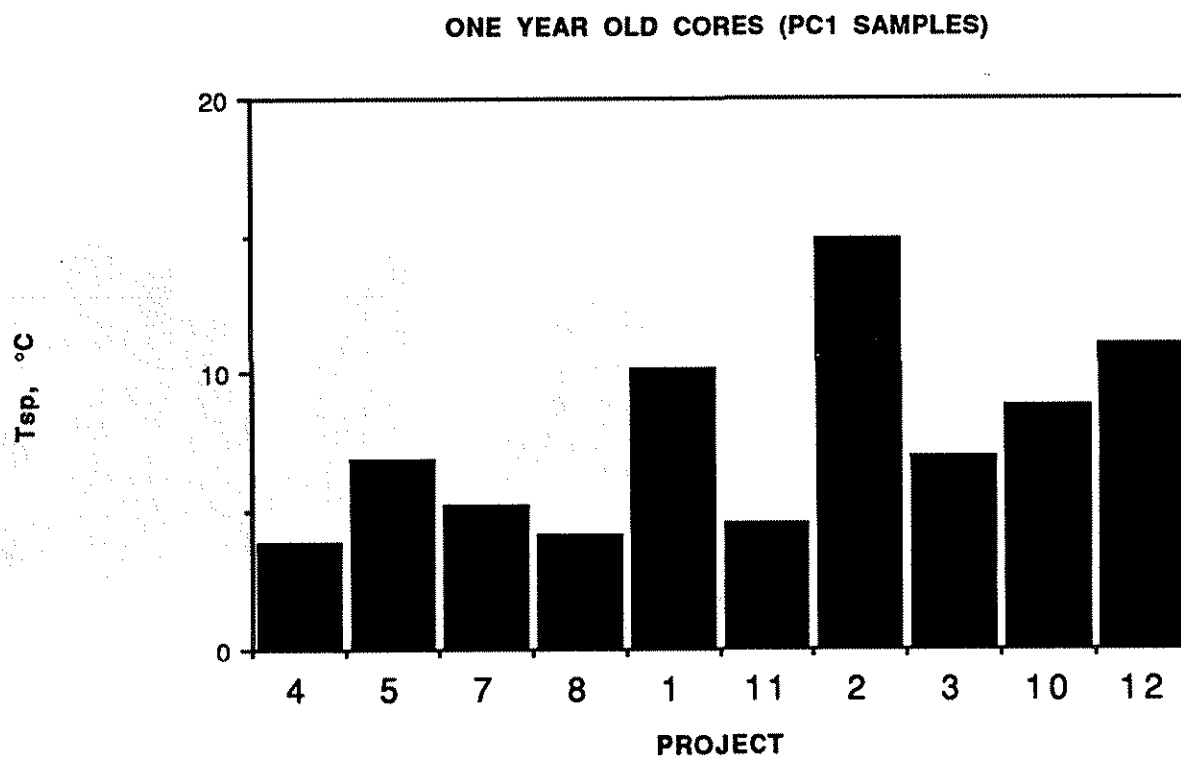


Figure 12. Softening temperatures (T<sub>sp</sub>), one-year old cores.

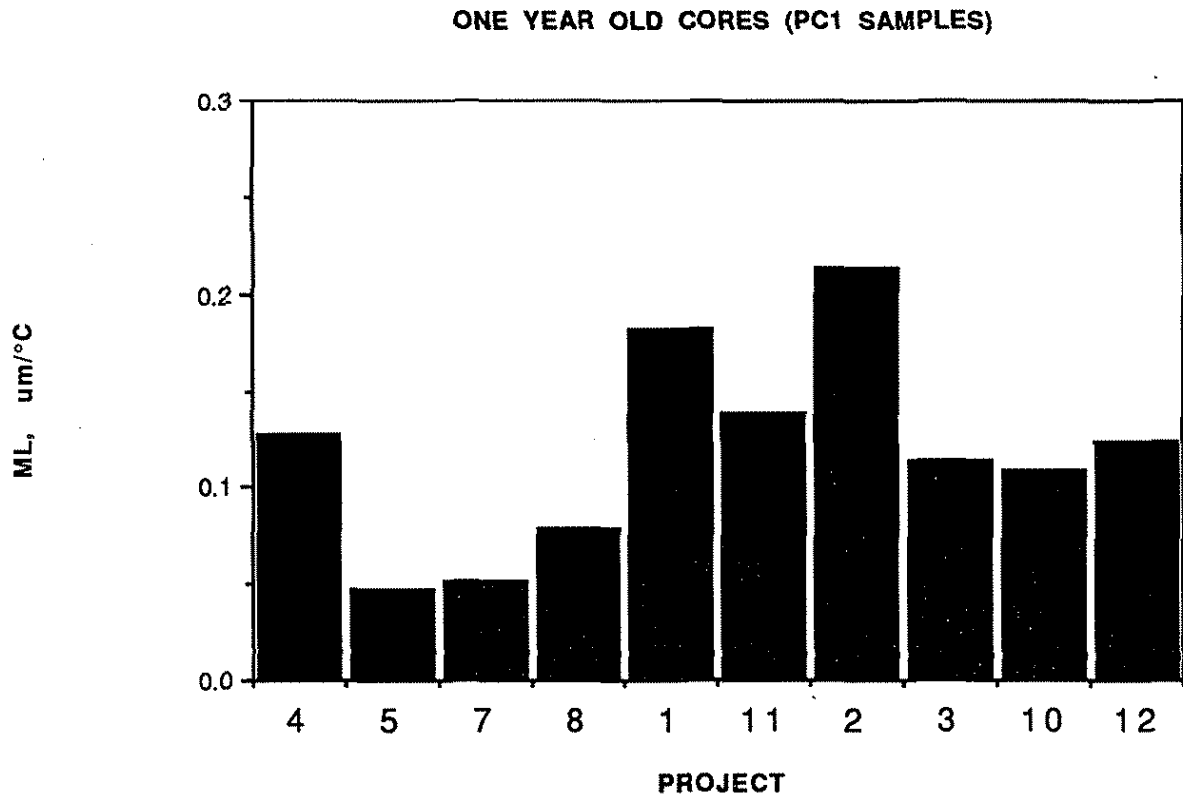


Figure 13. Low-temperature expansion slopes (ML), one-year old cores.

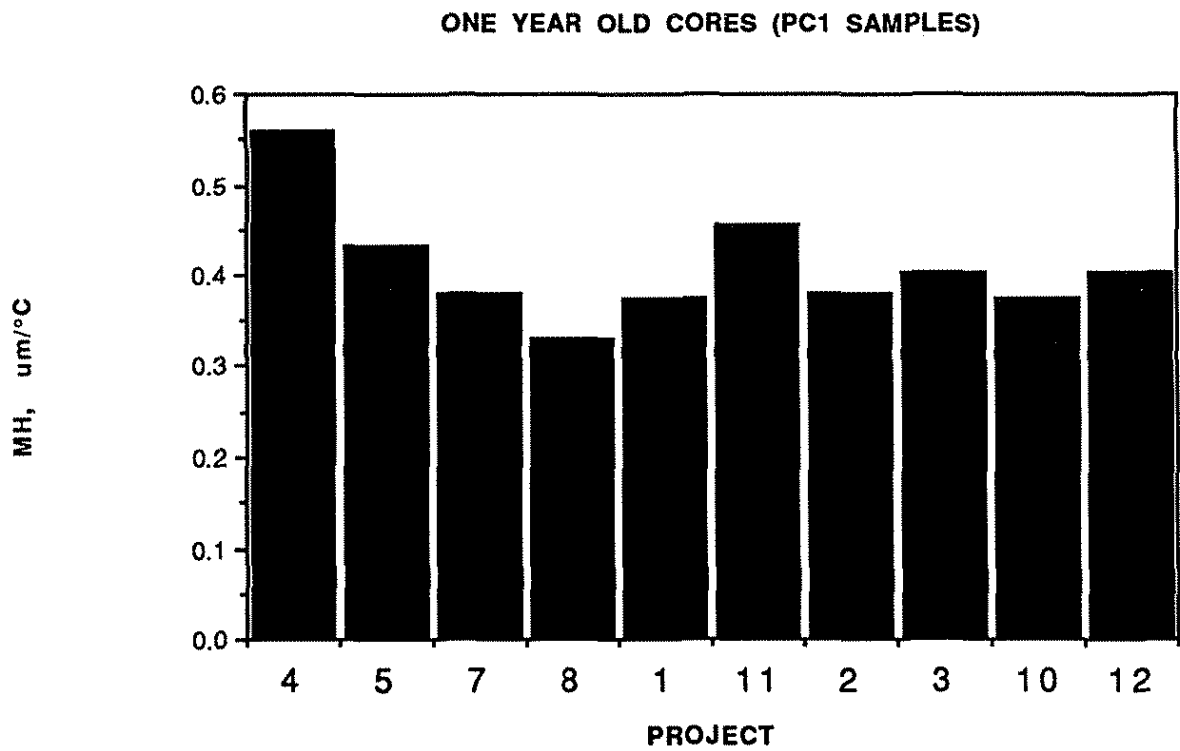


Figure 14. Intermediate temperature expansion slopes (MH), one-year old cores.

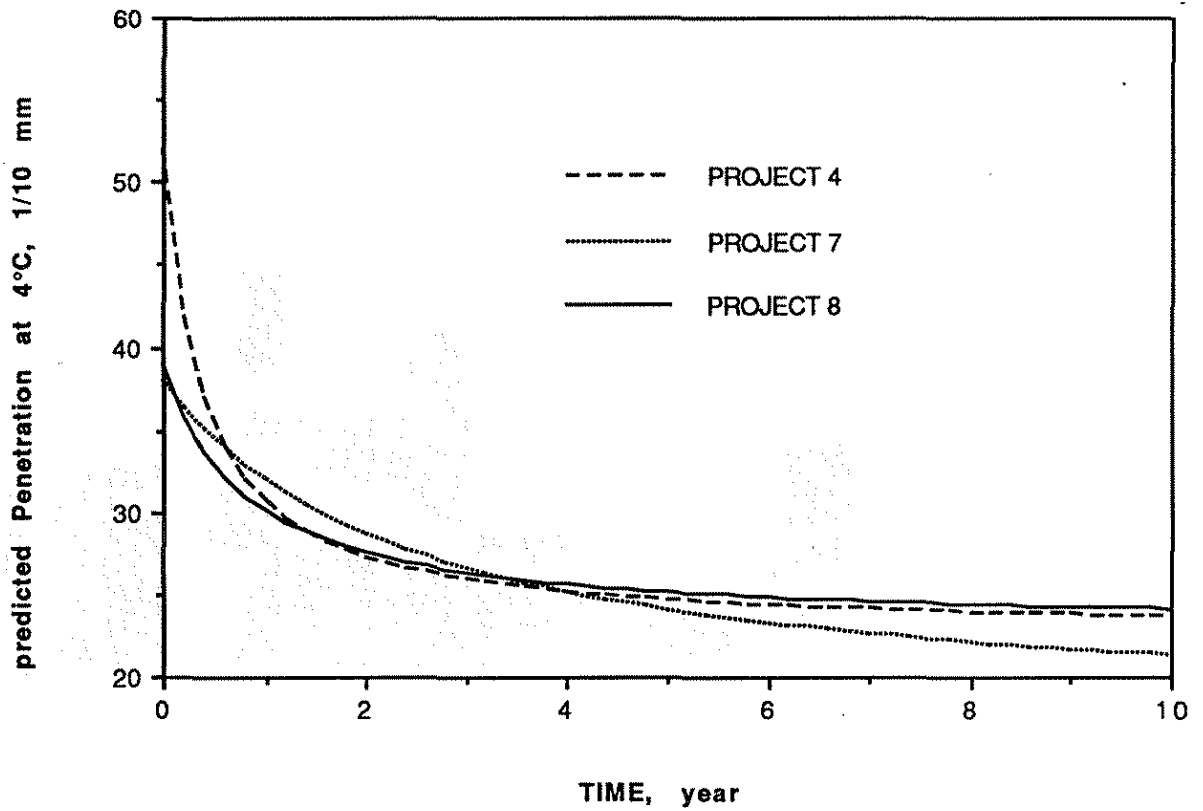


Figure 15. Predictive penetration at 4°C vs time.

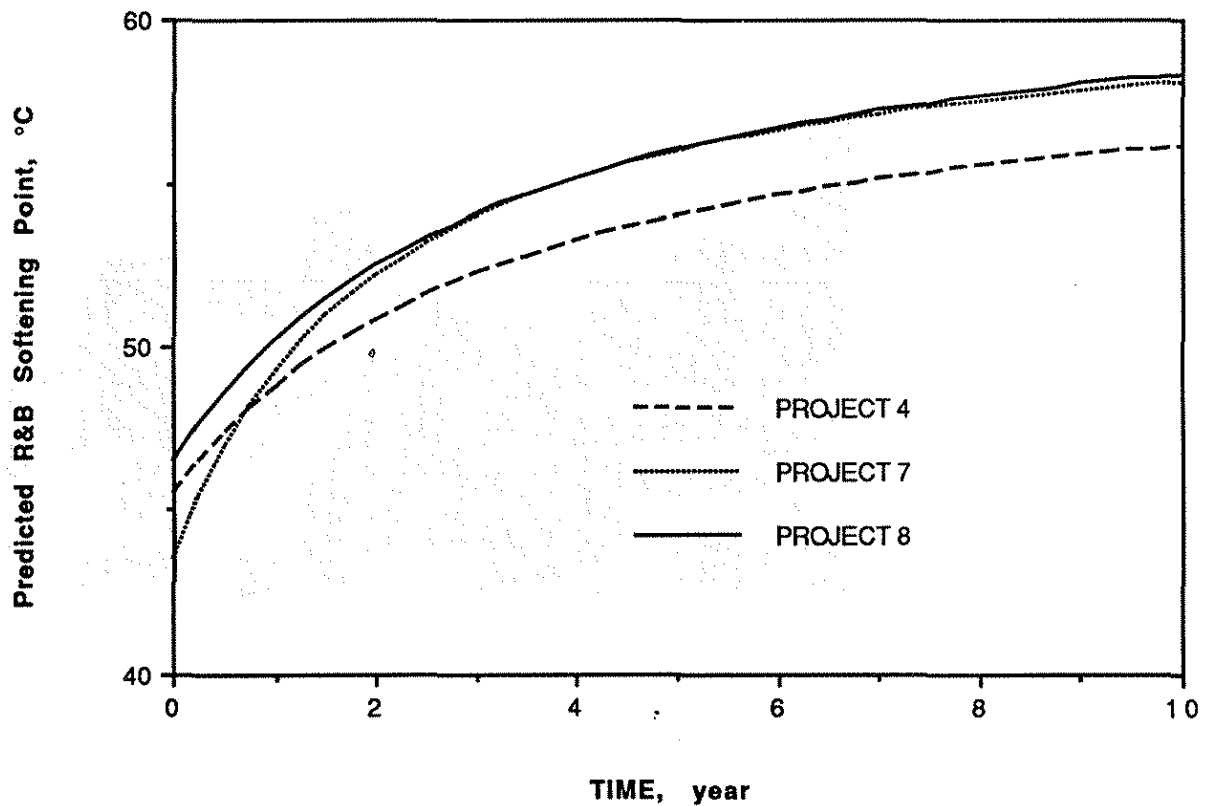


Figure 16. Predictive R&B softening point vs time.



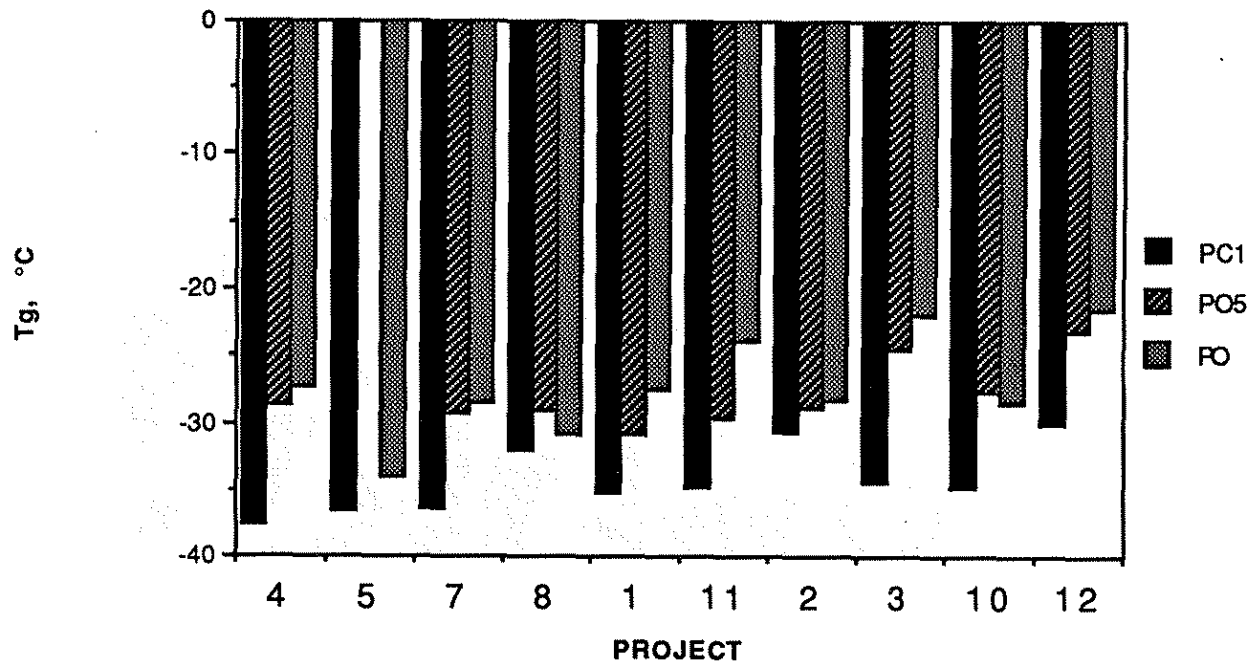


Figure 17. Glass transition temperatures ( $T_g$ ), PC1, PO5 and PO samples.

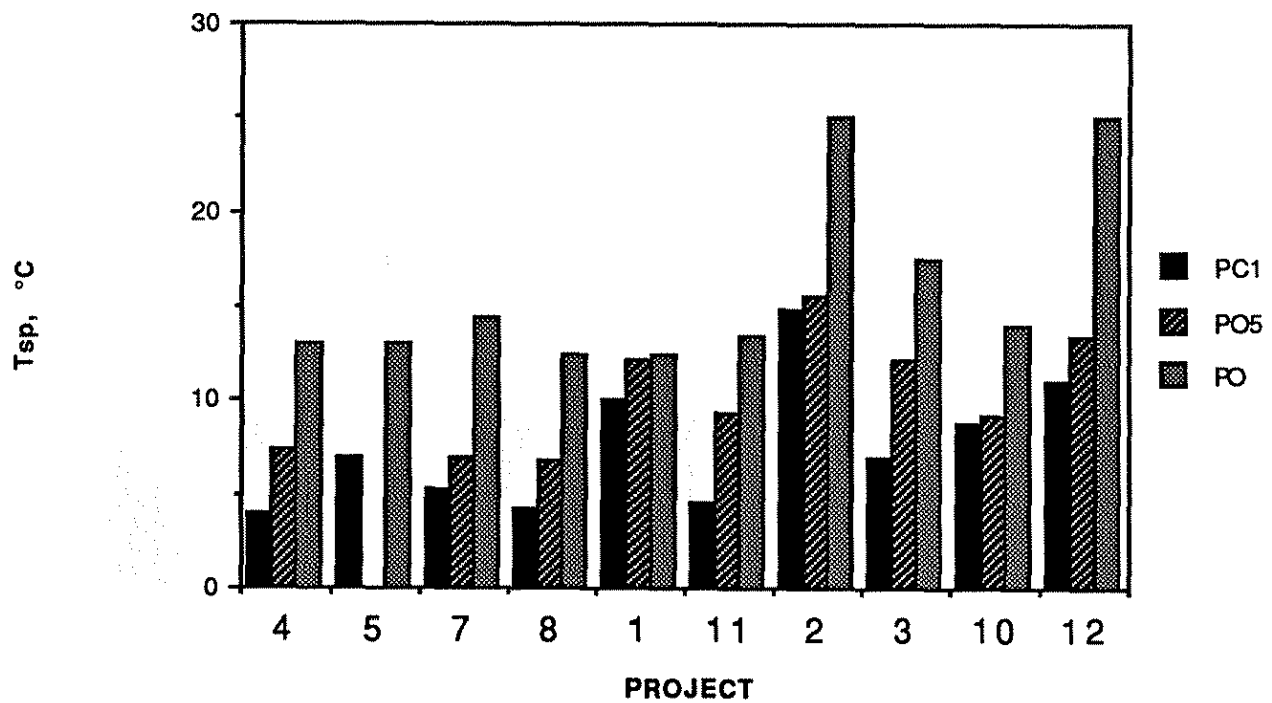


Figure 18. Softening temperatures ( $T_{sp}$ ), PC1, PO5 and PO samples.

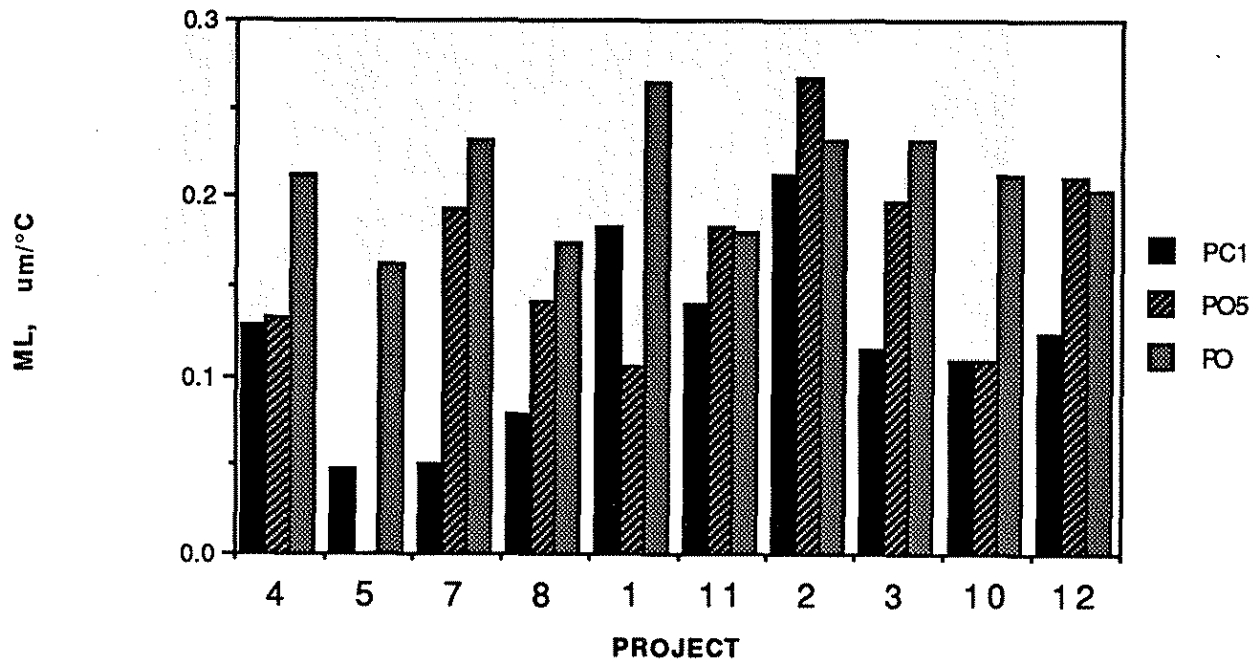


Figure 19. Low-temperature expansion slope (ML), PC1, PO5 and PO samples.

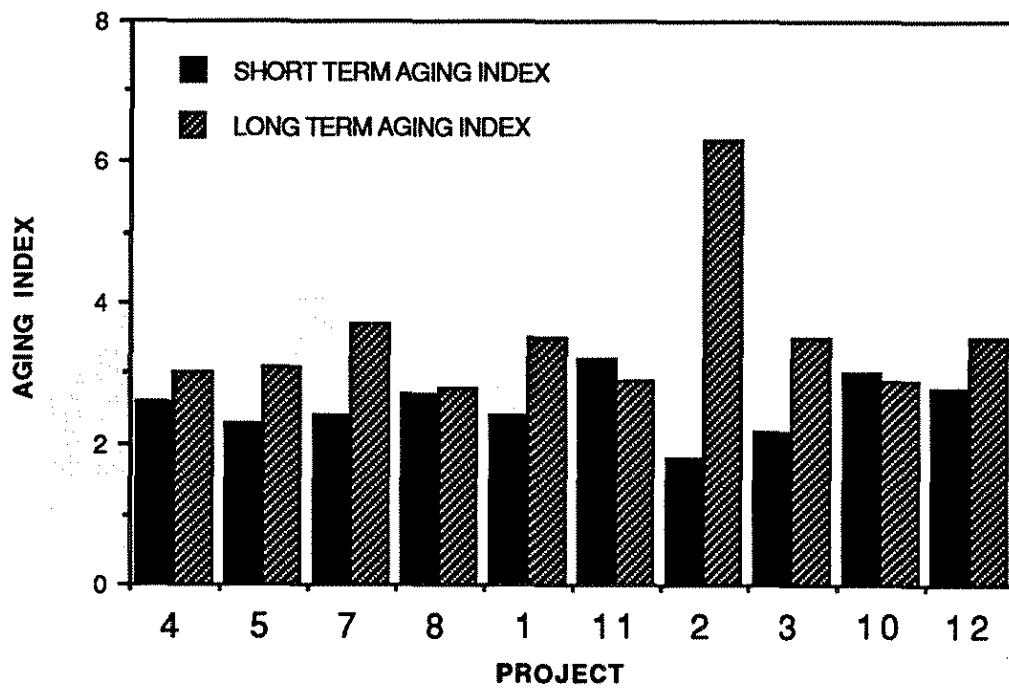


Figure 20. Short-term and long-term aging indices.

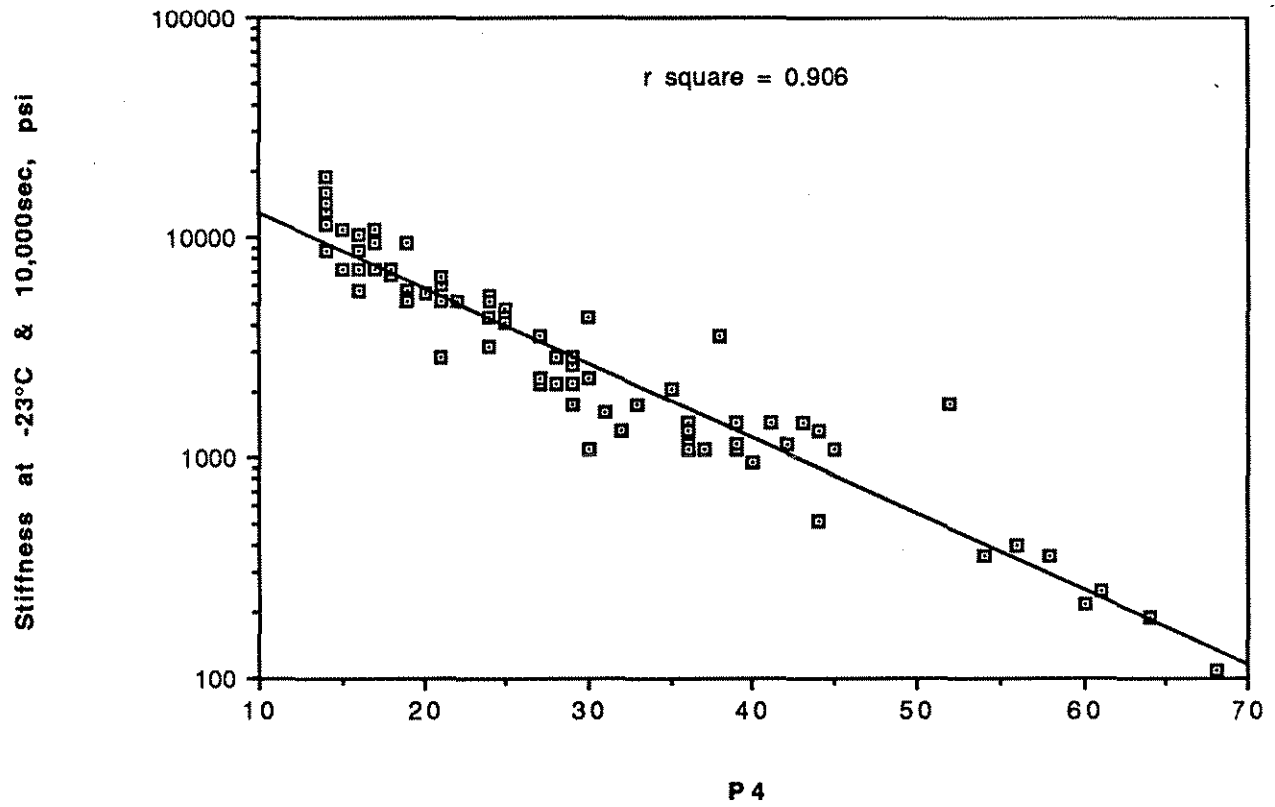


Figure 21. Stiffness at -23°C vs penetration at 4°C.

**APPENDIX I:**  
**Regression Coefficients Matrix**

APPENDIX I. Regression coefficient matrix.

	P5	P25	P4	R&B SP	VIS25
Intercept	3.76E+02	7.19E+03	1.79E+03	-5.54E+02	4.81E+08
X1	-5.61E+00	-1.05E+02	-2.48E+01	1.23E+01	-5.88E+05
X2	-1.29E+00	-3.05E+01	-8.17E+00	-2.53E+00	-1.00E+07
X3	-4.46E+00	-1.11E+02	-2.38E+01	1.60E+01	1.79E+05
X4	-9.87E-01	-4.11E+01	-1.22E+01	-4.81E+00	-1.33E+07
X5	-7.80E+00	-4.53E+01	-1.53E+01	9.73E-01	-5.76E+06
X6	-9.07E+00	-1.79E+02	-4.98E+01	2.67E+01	1.06E+07
X7	9.03E+00	6.30E+01	3.27E+01	-1.44E+01	-2.04E+07
X8	-6.73E+00	-7.38E+01	-2.40E+01	8.01E+00	-1.21E+06
MWT	1.91E-03	6.51E-02	1.46E-02	-4.66E-03	2.59E+03
POLYIDX	4.35E-02	-1.95E+01	-5.49E+00	-1.96E+00	-5.56E+06
Tg	-6.94E-02	6.80E-01	1.82E-02	-2.63E-01	-2.75E+04
Tsp	-1.71E-01	-1.94E+00	-7.46E-01	4.95E-01	4.03E+05
ML	-1.58E+01	-2.88E+02	-7.20E+01	4.59E+01	2.34E+07
MH	1.23E+01	1.64E+02	4.79E+01	-2.60E+01	-1.42E+07
	CF	SI	VIS60	VIS135	PR
Intercept	-8.79E-01	5.40E+00	-7.89E+05	-2.26E+04	-7.23E+00
X1	-7.25E-02	3.31E-02	1.22E+04	3.81E+02	1.78E-01
X2	1.55E-01	-1.86E-01	1.93E+03	-8.14E+01	-4.31E-02
X3	-1.79E-01	1.25E-01	1.77E+04	7.44E+02	2.39E-01
X4	2.96E-01	-2.97E-01	-7.66E+03	-3.51E+02	-8.88E-02
X5	-6.26E-02	-4.65E-03	2.14E+03	2.14E+01	-3.30E-02
X6	-1.45E-01	1.18E-01	3.32E+04	1.05E+03	3.07E-01
X7	2.95E-01	-2.95E-01	-9.83E+03	-6.63E+02	-1.24E-02
X8	-9.97E-02	4.52E-02	8.85E+03	5.14E+02	4.30E-02
MWT	-4.15E-05	6.80E-05	7.80E+00	3.49E-01	-6.01E-05
POLYIDX	1.21E-01	-1.30E-01	-9.62E+03	-3.66E+02	-5.18E-02
Tg	1.01E-02	-9.80E-03	-2.97E+02	-2.43E+00	-3.93E-03
Tsp	-5.29E-03	6.07E-03	8.17E+02	1.80E+01	3.78E-03
ML	-8.76E-01	6.78E-01	7.82E+04	1.64E+03	6.47E-01
MH	3.50E-01	-3.28E-01	-4.60E+04	-9.90E+02	-2.85E-01

	PI	CN	VTS	PVN60	PVN135
Intercept	-8.23E-01	1.57E+03	-1.41E+01	-5.26E+01	-2.46E+00
X1	3.69E-01	-1.72E+01	2.28E-01	5.00E-01	-8.38E-02
X2	-6.42E-01	-1.56E+01	1.92E-01	5.30E-01	-1.96E-02
X3	1.06E+00	-2.28E+01	1.16E-01	9.90E-01	5.02E-01
X4	-1.05E+00	5.28E+00	5.13E-02	-5.45E-01	-5.30E-01
X5	-4.56E-01	-1.51E+01	2.18E-01	6.07E-01	-4.85E-02
X6	6.83E-01	-3.84E+01	4.57E-01	1.16E+00	2.59E-02
X7	3.76E-02	6.30E-01	9.67E-03	8.34E-02	-5.68E-02
X8	-7.71E-02	-1.81E+01	1.06E-01	7.88E-01	3.70E-01
MWT	-6.81E-05	-8.25E-03	-6.05E-05	7.23E-04	6.27E-04
POLYIDX	-2.78E-01	7.59E+00	-6.28E-03	-5.14E-01	-3.59E-01
Tg	-4.48E-02	3.76E-02	-3.40E-03	1.00E-03	9.44E-03
Tsp	2.35E-02	-1.51E-01	4.32E-03	2.34E-03	-8.61E-03
ML	5.31E-01	-4.79E+01	9.50E-01	8.97E-01	-1.20E+00
MH	-1.43E-01	3.52E+01	-5.86E-01	-7.25E-01	5.38E-01

	CT	TES	S23	S29
Intercept	-1.25E+02	-1.37E+03	2.69E+02	6.99E+02
X1	7.38E-01	1.70E+01	-4.03E-01	-4.22E+00
X2	1.51E+00	9.81E+00	-6.43E+00	-1.31E+01
X3	-2.90E+00	1.39E+01	-2.53E-01	-3.57E+00
X4	1.34E+00	1.34E+01	-4.66E+00	-7.88E+00
X5	1.12E+01	1.13E+01	-1.01E+00	-1.10E+00
X6	-3.34E+00	2.83E+01	2.38E+00	-4.84E+00
X7	-6.54E+00	-1.14E+01	-1.22E+01	-2.01E+01
X8	6.56E+00	1.99E+01	6.32E-03	-2.25E+00
MWT	3.05E-03	-2.21E-03	4.60E-04	2.10E-03
POLYIDX	-1.83E+00	-1.03E+00	-2.01E+00	-3.03E+00
Tg	3.26E-01	2.61E-01	9.75E-02	2.02E-01
Tsp	-7.60E-02	3.50E-02	1.90E-01	2.49E-01
ML	-1.26E+01	2.01E+01	1.70E+01	3.71E+01
MH	-9.09E-01	-1.50E+01	-8.69E+00	-1.43E+01

**APPENDIX II:****Predicted vs. Measured Properties**

